



ARL-TR-7244 • MAR 2015



Verification and Demonstration for Transition of Non-Hexavalent Chromium, Low-VOC Alternative Technologies to Replace DOD-P-15328 Wash Primer for Multimetal Applications

by John Kelley, Thomas Considine, and Thomas Braswell

Approved for public release; distribution is unlimited.

NOTICES

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Citation of manufacturer's or trade names does not constitute an official endorsement or approval of the use thereof.

Destroy this report when it is no longer needed. Do not return it to the originator.



Verification and Demonstration for Transition of Non-Hexavalent Chromium, Low-VOC Alternative Technologies to Replace DOD-P-15328 Wash Primer for Multimetall Applications

by John Kelley, Thomas Considine, and Thomas Braswell
Weapons and Materials Research Directorate, ARL

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188		
<p>Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.</p> <p>PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.</p>					
1. REPORT DATE (DD-MM-YYYY) March 2015		2. REPORT TYPE Final		3. DATES COVERED (From - To) October 2009–September 2014	
4. TITLE AND SUBTITLE Verification and Demonstration for Transition of Non-Hexavalent Chromium, Low-VOC Alternative Technologies to Replace DOD-P-15328 Wash Primer for Multimetal Applications			5a. CONTRACT NUMBER		
			5b. GRANT NUMBER		
			5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S) John Kelley, Thomas Considine, and Thomas Braswell			5d. PROJECT NUMBER W12AR09		
			5e. TASK NUMBER		
			5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) US Army Research Laboratory ATTN: RDRL-WMM-C Aberdeen Proving Ground, MD 21005-5069			8. PERFORMING ORGANIZATION REPORT NUMBER ARL-TR-7244		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)			10. SPONSOR/MONITOR'S ACRONYM(S)		
			11. SPONSOR/MONITOR'S REPORT NUMBER(S)		
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT It has been known for quite some time that chemical treatments containing hexavalent chromium [Cr(VI)] are risks to both human health and the environment. In April of 2009, a memo was released from the Office of the Secretary of Defense (OSD) that specifically directs the military to approve the use of alternatives to Cr(VI) where they can perform adequately for the intended application and operating environment. Effectively, the memo directs Department of Defense (DOD) Military Departments to restrict the use of Cr(VI) unless no cost-effective alternative with satisfactory performance can be identified. This effort examines the effectiveness of alternative spray applied pretreatments for steel substrates versus DOD-P-15328 chromated wash primer. Eight nonchromate alternative candidates were evaluated against the baseline wash primer and untreated controls on multiple metal substrates, steel, galvanized steel, stainless steel, and aluminum (Al). The laboratory results identified 3 promising alternatives to the DOD-P-15328 with 1 outperforming the baseline chromate wash primer.					
15. SUBJECT TERMS DOD-P-15328, nonchromate, pretreatment, wash primer					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	18. NUMBER OF PAGES 65	19a. NAME OF RESPONSIBLE PERSON John Kelley
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			19b. TELEPHONE NUMBER (Include area code) 410-306-0837

Contents

List of Figures	v
List of Tables	vii
1. Introduction	1
2. Experimental Procedure	2
2.1 Sample Preparation	2
2.2 Wet Tape Adhesion	6
2.3 Dry Tape Adhesion	7
2.4 Pencil Hardness	7
2.5 Chip Resistance Test	8
2.6 Accelerated Corrosion Testing	9
2.7 Outdoor Exposure Testing at Cape Canaveral Air Force Station (CCAFS)	11
2.8 Performance Objectives	12
3. Results and Discussion	13
3.1 Wet Tape Adhesion	13
3.2 Dry Tape Adhesion	14
3.3 Pencil Hardness	16
3.4 Chip Resistance Testing	17
3.5 Neutral Salt Fog Corrosion (ASTM B117)	20
3.6 Accelerated Cyclic Corrosion (GM 9540P)	24
3.7 Outdoor Exposure	35
4. Limited Scale Demonstration	40
Mine-Resistant Ambush Protected (MRAP) Vehicle Doors	40
5. Conclusions	43
6. References	45

Appendix A. Observed Corrosion Rate (mils/year) at CCAFS Since 2011	47
Appendix B. Weather Data for 2012–13 at CCAFS Site	49
Appendix C. Weather Data for 2013–14 at CCAFS Site	51
List of Symbols, Abbreviations, and Acronyms	53
Distribution List	55

List of Figures

Fig. 1	Multimetal galvanic test specimen prior to pretreatment and paint.....	2
Fig. 2	(Left) Pretreatment application; (Right) Coating application at the ATC 5	
Fig. 3	ASTM D3359 Method B: cross hatch adhesion ratings	7
Fig. 4	Pencil hardness scale.....	8
Fig. 5	(Left) Example of the Q-Lab Gravelometer used to measure chip resistance per SAE J400; (Right) The area of a panel evaluated	8
Fig. 6	(Left) The chamber used to conduct the neutral salt fog ASTM B 117; (Right) The chamber used to conduct General Motors (GM) 9540P tests	10
Fig. 7	Satellite image of CCAFS/TACOM outdoor exposure site in relation to ocean	11
Fig. 8	Example of racks on CCAFS/TACOM outdoor exposure site.....	12
Fig. 9	Bonderite 7400 on (from left to right) Al, CRS, galvanized, and stainless steel (53030 top, 53022 bottom)	18
Fig. 10	Ecosil 5-1 on (from left to right) Al, CRS, galvanized, and stainless steel (53030 top, 53022 bottom)	18
Fig. 11	DOD-P-15328 wash primer on (from left to right) Al, CRS, galvanized, and stainless steel (53030 top, 53022 bottom).....	19
Fig. 12	Picklex on (from left to right) Al, CRS, galvanized, and stainless steel (53030 top, 53022 bottom)	19
Fig. 13	Bonderite 7400 rated a 7.4 on Al panels with MIL-DTL-53022 at 672 h.....	22
Fig. 14	DOD-P-15328 rated a 7.2 on Al panels with MIL-DTL-53022 at 672 h.....	22
Fig. 15	Picklex rated a 0.0 on Al panels with MIL-DTL-53022 at only 72 h.....	22
Fig. 16	Bonderite 7400 on multimetal panels with MIL-DTL-53022 after 672 h.....	24
Fig. 17	DOD-P-15328 on multimetal panels with MIL-DTL-53022 after 672 h	24
Fig. 18	Bonderite 7400 on CRS primed with MIL-DTL-53022 after 80 cycles.	26
Fig. 19	Bonderite 7400 on CRS primed with MIL-DTL-53030 after 80 cycles.	26
Fig. 20	Ecosil 5-1 on CRS primed with MIL-DTL-53022 after 80 cycles	26
Fig. 21	Ecosil 5-1 on CRS primed with MIL-DTL-53030 after 80 cycles	27
Fig. 22	DOD-P-15328 on CRS primed with MIL-DTL-53022 after 80 cycles..	27
Fig. 23	DOD-P-15328 on CRS primed with MIL-DTL-53022 after 80 cycles..	27

Fig. 24	Bonderite 7400 on Al primed with MIL-DTL-53022 after 80 cycles	29
Fig. 25	Bonderite 7400 on Al primed with MIL-DTL-53030 after 80 cycles	29
Fig. 26	Ecosil 5-1 on Al primed with MIL-DTL-53022 after 80 cycles.....	29
Fig. 27	Ecosil 5-1 on Al primed with MIL-DTL-53030 after 80 cycles.....	30
Fig. 28	DOD-P-15328 on Al primed with MIL-DTL-53022 after 80 cycles	30
Fig. 29	DOD-P-15328 on Al primed with MIL-DTL-53030 after 80 cycles	30
Fig. 30	NCP nonchromate wash primer multimetal panels delaminating after 3 cycles.....	32
Fig. 31	Bonderite 7400 on multimetal panels primed with MIL-DTL-53022 after 80 cycles.....	32
Fig. 32	Bonderite 7400 on multimetal panels primed with MIL-DTL-53030 after 80 cycles.....	33
Fig. 33	Ecosil 5-1 on multimetal panels primed with MIL-DTL-53022 after 80 cycles.....	33
Fig. 34	Ecosil 5-1 on multimetal panels primed with MIL-DTL-53030 after 80 cycles.....	34
Fig. 35	DOD-P-15328 on multimetal panels primed with MIL-DTL-53022 after 80 cycles.....	34
Fig. 36	DOD-P-15328 on multimetal panels primed with MIL-DTL-53030 after 80 cycles.....	35
Fig. 37	Bonderite 7400 on CRS primed with MIL-DTL-53022 after 2 years	36
Fig. 38	Bonderite 7400 on CRS primed with MIL-DTL-53030 after 2 years	37
Fig. 39	DOD-P-15328 on CRS primed with MIL-DTL-53022 after 2 years	37
Fig. 40	DOD-P-15328 on CRS primed with MIL-DTL-53030 after 2 years	37
Fig. 41	Picklex on Al primed with MIL-DTL-53022 after 1 year	38
Fig. 42	Bonderite 7400 on Al primed with MIL-DTL-53022 after 2 years.....	39
Fig. 43	Bonderite 7400 on Al primed with MIL-DTL-53030 after 2 years.....	39
Fig. 44	DOD-P-15328 on Al primed with MIL-DTL-53022 after 2 years	39
Fig. 45	DOD-P-15328 on Al primed with MIL-DTL-53030 after 2 years	39
Fig. 46	(Left) MRAP door being treated with Oxsilan 9810/2; (Right) Painted doors on outdoor exposure racks at ARL	41
Fig. 47	MRAP door with Oxsilan 9810/2 after 2 years with close-up of scribed area.....	42
Fig. 48	MRAP door with DOD-P-15328 WP after 2 years with close-up of scribed area	43

List of Tables

Table 1	Project test matrix	4
Table 2	Candidate names and technology description.....	5
Table 3	Film thicknesses for full coating systems	6
Table 4	SAE J400 ratings for number of chips in a 4 inch × 4 inch area	9
Table 5	SAE J400 ratings for size of chips in a 4 inch × 4 inch area	9
Table 6	Cycle details for the GM 9540P cyclic corrosion test	10
Table 7	Performance objectives with success criteria	13
Table 8	Wet tape adhesion ratings (ASTM D3359 Method A)	14
Table 9	Dry tape adhesion ratings (ASTM D3359 Method B).....	15
Table 10	Pencil hardness ratings (ASTM D3363)	16
Table 11	Chip resistance ratings per SAE J400	17
Table 12	ASTM D1654 ratings for ASTM B117 neutral salt fog testing on CRS	20
Table 13	ASTM D1654 ratings for ASTM B117 neutral salt fog testing on Al.....	21
Table 14	ASTM D1654 ratings for ASTM B117 salt fog testing on multimetal panels	23
Table 15	ASTM D1654 ratings for GM 9540P cyclic corrosion testing on CRS	25
Table 16	ASTM D1654 ratings for GM 9540P cyclic corrosion testing on Al....	28
Table 17	ASTM D1654 ratings for GM9540P testing on “multimetal” specimens	31
Table 18	ASTM D1654 ratings for outdoor exposure testing on CRS panels.....	36
Table 19	ASTM D1654 ratings for outdoor exposure testing on Al panels	38
Table 20	ASTM D1654 ratings for outdoor exposure testing on multimetal panels	40
Table 21	ASTM D1654 ratings for outdoor exposure testing on MRAP doors ...	43

1. Introduction

Under Army Regulation (AR) 750-1,¹ all Army-based ground equipment is required to have a full Chemical Agent Resistant Coating (CARC) system. The description of what typically comprises a full CARC system is defined in MIL-DTL-53072.² The typical CARC system consists of: a) conversion coating or pretreatment in direct contact with the substrate properly applied in accordance with (IAW) TT-C-490,³ followed by b) epoxy primer IAW MIL-DTL-53022⁴ or MIL-DTL-53030,⁵ and c) the polyurethane-based topcoat IAW MIL-DTL-53039⁶ or MIL-DTL-64159.⁷ Whole platforms and vehicles instead will require the spray applied wash primer, DOD-P-15328,⁸ as the pretreatment prior to applying the epoxy primer and polyurethane topcoat.

The DOD-P-15328 chromated wash primer was developed by the Bakelite Company during World War II while under contract by the US Government. The development of the wash primer was motivated by an urgent need for a substitute metal pretreatment in ship construction. It is a 2-component system consisting of a zinc chromate rust inhibiting pigment in a flexible adhering polymer activated by phosphoric acid prior to use. The DOD-P-15328 wash primer has been a workhorse pretreatment for the Department of Defense (DOD), performing adequately for many years to passivate metal surfaces, provide protection against corrosion, and improve the adhesion of the primer to the substrate/pretreatment.⁹

It has been known for decades that chemical treatments containing hexavalent chromium are risks to both human health and the environment. In April of 2009, a memorandum was released from the Office of the Secretary of Defense (OSD) that outlined a new policy for reducing the use of hexavalent chromium [Cr(VI)] for DOD applications. The memo specifically directs the military to approve the use of alternatives to Cr(VI) where they can perform adequately for the intended application and operating environment, update relevant technical documents and specifications to authorize the use of qualified alternatives to Cr(VI), and requires the Program Executive Office (PEO) or equivalent, in coordination with the Military Department's Corrosion Control and Prevention Executive (CCPE), to certify that there is no acceptable alternative to the use of Cr(VI) on a new system. Effectively, the memo directs DOD Military Departments to restrict the use of Cr(VI) unless no cost-effective alternative with satisfactory performance can be identified.¹⁰

This effort will examine the effectiveness of alternative spray applied pretreatments for multiple metal substrates to include steel, galvanized steel, stainless steel and aluminum (Al) versus the baseline DOD-P-15328 chromated wash primer. The alternatives that meet performance requirements of Federal specification TT-C-490F will be listed on the qualified product database (QPD) for the specification. The QPD gives users the option to use these nonchromate alternatives without requiring drawing changes, effectively expediting the implementation of the alternative products.

2. Experimental Procedure

2.1 Sample Preparation

Standard test coupons of 3 main sizes were used for testing the alternatives: 4 inch \times 6 inch \times 3/16 inch, and 4 inch \times 12 inch \times 1/8 inch. These standard flat test coupons were made from 4 alloys: Al 6061, ASTM A1008¹¹ cold rolled steel (CRS), galvanized steel, and SAE 304 stainless steel.¹² In addition to these standard test coupons, a special galvanic multimetal test panel was fabricated using the 4 inch \times 12 inch \times 3/16 inch CRS U-weld panels as a base. These panels have a steel “U” channel, welded in the center of the bottom quarter of the CRS panel with the “U” channel’s concave side facing upward. An Al 6061 “L” bracket was fastened to the top quarter of the CRS panel using 1 stainless steel screw and washer and 1 galvanized steel screw and washer. After assembly, the Al “L” bracket was masked off with tape and the entire multimetal panel was abrasive blasted using 60-grit Al oxide blast media to a surface profile of 1.5 mil. An example of a multimetal test panel can be seen in Fig. 1. The rest of the panels were not abrasive blasted and retained a mill finish.

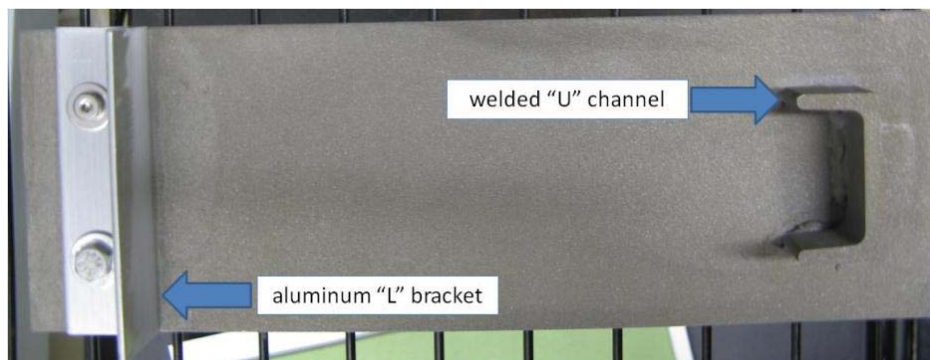


Fig. 1 Multimetal galvanic test specimen prior to pretreatment and paint

Table 1 shows the test matrix for the project. The preliminary screening testing performed to determine if each pretreatment application parameters are within that of the standard wash primer is not reflected in the matrix. The goal is to identify a replacement for chromate wash primer that does not significantly add to the overall processing time. The standard DOD-P-15328 wash primer used as the baseline in this program was manufactured by the Sherwin-Williams Company.

DOD-P-15328 application criteria:

- Mix ratio: 4 parts Comp A and 1 part Comp B
- Thin admixed material with 4 parts isopropyl alcohol
- Application is 0.3–0.5 dry mils
- Epoxy primer applied after complete drying—30–60 min.

Using the manufacturer's recommended parameters, steel test coupons were sprayed to assure that preparation and pretreatment time prior to painting was similar to the standard wash primer. Those that passed screening were included in the test matrix and a brief description of each candidate technology can be seen in Table 2.

Table 1 Project test matrix

Pretreatment	Primer	Topcoat	Wet and Dry Cross Hatch Adhesion				Pencil Hardness ASTM D3363				SAE J400 Gravelometer				GM9540P		Outdoor Weathering		ASTM B117		Humidity Chamber	ASTM B117	GM 9540P	Outdoor Weathering
			4"x12"x1/8"				4"x12"x1/8"				4"x6"x3/16"				4x6x3/16"		4x6x3/16"		4x6x3/16"		Special multi-metal galvanic test panel			
			CRS 1008	SS 304	GAL	AI 6061	CRS 1008	SS 304	GAL	AI 6061	CRS 1008	SS 304	GAL	AI 6061	6061	CRS 1008	6061	CRS 1008	6061	CRS 1008				
Picklelex 20	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022 T2	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
SurTec 650	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
AC-10	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
PPG 11-TGL-07-Z	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
Oxilan 9810/2	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
Bonderite 7400	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
ECO 5-1	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
NCP non-chromate Wash Primer	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
DOD-P-15328 Standard Wash Primer	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
No Pretreatment	53030	53039																				5	5	5
	53022	53039																				5	5	5
	none	none																			3			

Table 2 Candidate names and technology description

Pretreatment Name	Technology Description
Picklex 20	Water-based conversion coating containing less than 10% Phosphoric acid by volume
SurTec 650	Trivalent chrome direct-to-metal (DTM) pretreatment designed for Al, but has had success on steel substrates in Environmental Security Technology Certification Program (ESTCP) Weapons Platform (WP) 200906 project
Aero-Green AC-10	Rust remover, metal prep. Water reducible phosphoric acid blend
PPG 11-TGL-07-Z	Zirconium-based pretreatments similar to Zircobond 4200 that has been reformulated for spray application
Oxsilan 9810/2	Chemetall spray applied version of a silane pretreatment evaluated in ESTCP WP 200906 project
Bonderite 7400	Henkel Bonderite is a spray-in-place application nano technology with phosphoric acid
Ecosil Eco 5-1	Technology-Nano-size complex organic-inorganic material
NCP WP N-8237-2.5 A/B	NCP Coatings, commercial-off-the-shelf (COTS) nonchromate, low-VOC wash primer

Pretreatments were applied IAW each manufacturer's process by US Army Research Laboratory (ARL) and Aberdeen Test Center (ATC) personnel. In some cases, a representative from the manufacturer was on hand to observe the application process. All pretreatments were spray applied per manufacturer's recommended procedures. The pretreatments were given adequate time to cure, after which the test coupons were painted by ATC personnel at the ATC paint shop under ARL supervision (Fig. 2). Test coupons were primed with either MIL-DTL-53030 Type IV or MIL-DTL-53022 Type II and top coated with MIL-DTL-53039 Type II. ARL recorded both dry and wet film thicknesses, which can be seen in Table 3.

**Fig. 2 (Left) Pretreatment application; (Right) Coating application at the ATC**

Table 3 Film thicknesses for full coating systems

Pretreatment	Coating System	Al	CRS	Galv	SS
Picklex 20	53030/53039	5.58	4.48	6.44	5.36
	53022/53039	5.24	6.68	5.60	7.18
SurTec 650	53030/53039	4.94	3.80	4.80	4.40
	53022/53039	5.66	5.56	5.74	6.68
Aero-Green AC-10	53030/53039	6.92	4.16	5.22	3.66
	53022/53039	5.86	5.40	4.30	6.74
PPG 11-TGL-07-Z	53030/53039	4.72	5.16	5.20	5.78
	53022/53039	6.44	6.64	5.56	8.72
Oxsilan 9810/2	53030/53039	4.44	5.88	4.78	4.28
	53022/53039	4.74	7.48	4.66	4.42
Bonderite 7400	53030/53039	3.9	5.52	4.34	5.66
	53022/53039	6.82	4.98	8.32	8.22
Ecosil Eco 5-1	53030/53039	4.14	5.32	4.52	4.48
	53022/53039	5.86	5.10	4.92	4.52
NCP WP N-8237-2.5 A/B	53030/53039	6.04	7.04	6.02	7.68
	53022/53039	8.80	8.72	6.18	8.20
DOD-P-15328 WP	53030/53039	4.26	4.40	4.64	5.68
	53022/53039	5.90	7.78	7.40	7.24

2.2 Wet Tape Adhesion

The wet tape adhesion test evaluates the coating's ability to resist penetration by water. This test is performed IAW Method 6301 of FED-STD-141¹³ (Paint, Varnish, Lacquer and Related Materials; Methods of Inspection Sampling and Testing) and rated per American Society for Testing and Materials (ASTM) D3359¹⁴ Method A. An "X" scribe is required on all test panels. Al, CRS, galvanized steel, and stainless steel were evaluated in the 24-h wet tape adhesion test. The samples were immersed in distilled water for 24 h at ambient temperature. The panels were then removed from the water and dried by wiping with a soft cloth. Two parallel lines are scribed approximately 1 inch apart with an "X" scribed between the 2 parallel lines ensuring that the coating has been scribed through to the substrate. A complete lap of tape is removed from the roll and discarded prior to removing the length of tape used for the test. Tape is removed from the roll at a constant, steady rate and cut to a length to completely cover the scribed area. The tape is then placed over the scribes and smoothed out by rolling with a 3-lb roller. The tape selected for this testing was 3M 250 Flatback Masking Tape, as approved by the CARC Commodity Manager and meeting the requirements of TT-C-490F. The tape is removed at a rapid, constant rate at an angle of approximately 180° to the surface. The areas around the scribes are inspected for peel-away/delamination and the unscribed immersed area is

inspected for blistering. Each panel is rated and photo-documented per ASTM D3359 Method A. To further ensure fairness, a second rating was made by a different ARL researcher. The 2 ratings were averaged together to create the final rating.

2.3 Dry Tape Adhesion

Tests were conducted at room temperature as defined in ASTM D3359 Method B. An area of the panel free of blemishes is selected. Using a sharp cutting tool, 6 parallel cuts at 2-mm spacing through the paint film to the metal substrate were made. A second series of cuts normal to the plane of the initial set were then made. Both cuts were made ensuring that they were sufficiently long enough to make a complete set of 6×6 grid lines. The grids were repeated in 15 other areas on test coupons. As only 10 data points per coupon were necessary, this gave some flexibility to the researchers to overcome the difficulty of creating uniform cross hatch grids. The grid lines are then brushed lightly to remove any detached flakes or ribbons of coating. A complete lap of tape is removed from the roll and discarded prior to removing the length of tape used for the test. A length of tape is removed at a steady (i.e., not jerked) rate and cut about 75-mm-long (3 inches). The center of the tape was placed over the grid and the area of the grid smoothed into place by a finger. To ensure good contact with the film, the tape was rubbed firmly with the eraser on the end of a pencil. The tape is then removed by seizing the free end and rapidly pulling (not jerked) back upon itself at as close to an angle of 180° to the surface of the panel as possible. Following the tape pull off, each grid is rated using the classification in ASTM D 3359 Method B, shown in Fig. 3. To further ensure accuracy, a second rating was made by a different ARL researcher. The 2 ratings were averaged to create the final rating.





Surface of cross-cut area from which flaking has occurred. (Example for 6 parallel cuts).	None					Greater than 65%
Classification	5	4	3	2	1	0

Fig. 3 ASTM D3359 Method B: cross hatch adhesion ratings

2.4 Pencil Hardness

A set of Staedtler Lumograph graphite drawing pencils was used to obtain pencil hardness values of the coating system. The coating system was allowed a full 7 days to cure in controlled laboratory conditions. Testing was done IAW ASTM D3363,¹⁵ “Standard Test Method for Film Hardness by Pencil Test” (Fig. 4). In

pencil hardness testing, the pencil is first prepared using a draftsman-type mechanical pencil sharpener to expose the cylinder of lead within the pencil. The lead is then placed normal to a piece of 400-grit sandpaper and ground to have a flat, chip-free surface. Starting with the hardest value pencil, in this case 4H, the operator holds the pencil at a 45° angle to the surface of the panel and pushes the pencil into the coating for 1/4 inch. If the pencil tip penetrates into the coating, the next softer pencil is selected and the test is redone until such a point that the pencil is unable to penetrate the coating. The hardness value of the pencil that is incapable of penetrating the coating is recorded as the pencil hardness value of the coating. The pencil hardness test can also expose catastrophic adhesion failures with respect to the coating and the substrate. This testing was done concurrently by 2 ARL researchers and their results were averaged to eliminate potential strength biases.

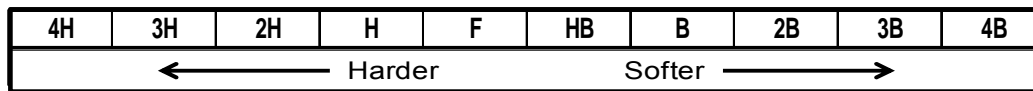


Fig. 4 Pencil hardness scale

2.5 Chip Resistance Test

Prior to beginning the tests, each panel was digitally photo-documented. The panels were then subjected to chip resistance testing IAW SAE J400¹⁶ at ambient temperature using a Q-Lab Gravelometer (Fig. 5 on left). The panels are held in a 45° angle specimen holder and air pressure is used to propel gravel at the sample. The test sample is then removed and gently wiped off with a clean cloth. Tape (3M #898 filament strapping tape as specified in SAE J400) is then applied to the entire tested surface to remove any loose fragments of the coating. The tested panel is then compared to standard SAE transparencies to determine a chipping rating (Fig. 5 on right).

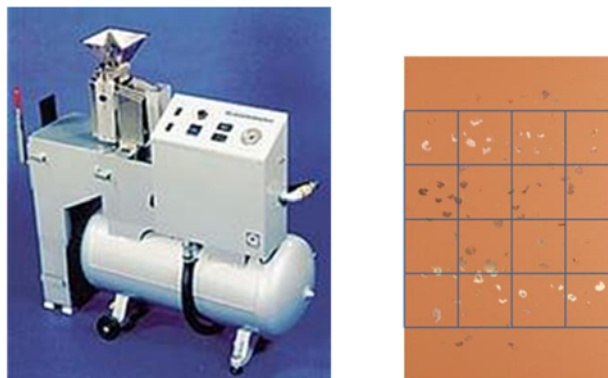


Fig. 5 (Left) Example of the Q-Lab Gravelometer used to measure chip resistance per SAE J400; (Right) The area of a panel evaluated

Panels were again digitally photographed following the tests and rated IAW SAE J400; ratings for each panel were recorded. The total number of chips inside a 4 inch \times 4 inch grid (16-square-inch area) using a transparency overlay was counted and the rating obtained using Table 4. The average size of the chips was measured and rated using Table 5. For panels without a dominant chip size, the second most prevalent chip size was included (for example, a “B/A” rating had at least 2/3 chips of size “B” and 1/3 chips of size “A”).

Table 4 SAE J400 ratings for number of chips in a 4 inch \times 4 inch area

SAE J400 Rating	10	9	8	7	6	5	4	3	2	1	0
No. of Chips in 4 \times 4 Grid	0	1	2–4	5–9	10–24	25–49	50–74	75–99	100–149	150–250	>250

Table 5 SAE J400 ratings for size of chips in a 4 inch \times 4 inch area

A	<1 mm	(approximately <0.03 inch)
B	1–3 mm	(approximately <0.03–0.12 inch)
C	3–6 mm	(approximately <0.12–0.25 inch)
D	>6 mm	(approximately <0.25 inch)

2.6 Accelerated Corrosion Testing

Two accelerated corrosion test chambers were used to evaluate the steel test panels. The Harshaw Model 22 for standard ASTM B117¹⁷ Neutral Salt Fog and an Atotech Model CCT-NC-30 for cyclic corrosion using GM9540P.¹⁸ Figure 6 shows both test chambers in the laboratory where the testing was carried out. Test panels in each corrosion test were “X” scribed, and rated IAW ASTM D1654¹⁹ except for the multimetal panels which only had a single diagonal scribe that was dictated by panel geometry. In each case, the test panels were scribed through the coating to the substrate. The samples were then placed in their respective chambers, leaning at an angle between 15° and 30° from the vertical with the scribed surface facing upwards.



Fig. 6 (Left) The chamber used to conduct the neutral salt fog ASTM B 117; (Right) The chamber used to conduct General Motors (GM) 9540P tests

The ASTM B117 neutral salt fog conditions are 95 °F with saturated humidity and atomized fog of 5% NaCl solution. The GM 9540P test consists of the 18 separate stages that are listed in Table 6 and include the following: saltwater spray, humidity, drying, ambient, and heated drying. Table 6 represents the environmental conditions and duration of each of the 18 stages in 1 complete GM9540P cycle. The standard 0.9% NaCl, 0.1% CaCl₂, 0.25% NaHCO₃ test solution was used. The cyclic chamber was verified with standard steel mass loss coupons as described in the GM 9540P test specification. The salt fog chambers were verified using temperature, pH and deposition rate measurements as per ASTM B117.

Table 6 Cycle details for the GM 9540P cyclic corrosion test

Interval	Description	Time (min)	Temperature (+3 °C)
1	Ramp to salt mist	15	25
2	Salt mist cycle	1	25
3	Dry cycle	15	30
4	Ramp to salt mist	70	25
5	Salt mist cycle	1	25
6	Dry cycle	15	30
7	Ramp to salt mist	70	25
8	Salt mist cycle	1	25
9	Dry cycle	15	30
10	Ramp to salt mist	70	25
11	Salt mist cycle	1	25
12	Dry cycle	15	30
13	Ramp to humidity	15	49
14	Humidity cycle	480	49
15	Ramp to dry	15	60
16	Dry cycle	480	60
17	Ramp to ambient	15	25
18	Ambient cycle	480	25

2.7 Outdoor Exposure Testing at Cape Canaveral Air Force Station (CCAFS)

CRS, Al 6061, and multimetal specimens were shipped from ARL to CCAFS for outdoor testing at the Tank Automotive and Armaments Command (TACOM) beach exposure site. Cape Canaveral is considered one of the most corrosive environments in the continental United States. The corrosion rate observed by ASTM is 5.17 mpy on standard steel mass loss coupons at 55 m inland.²⁰ For this reason, ARL has selected this outdoor exposure facility for much of its outdoor testing. The ARL corrosion racks are set at approximately 170 m inland and parallel to the ocean facing southeast (Fig. 7). The average corrosion rate in mpy observed by ARL since 2011 on standard mass loss coupons is 1.82 mpy at 170 m inland (see Appendix A). The test panels are scribed IAW ASTM D1654 and held in place on wood composite racks with nylon stand offs and stainless steel fasteners (Fig. 8). The coupons are being inspected and evaluated biannually in March and September IAW ASTM D1654 for both corrosion creep from the scribe as well as blistering in the field, as indicated by TT-C-490F.



Fig. 7 Satellite image of CCAFS/TACOM outdoor exposure site in relation to ocean



Fig. 8 Example of racks on CCAFS/TACOM outdoor exposure site

Weather data is collected using a data-logging weather station that is downloaded annually and can be seen in Appendixes B and C. The average rate of corrosion shown in Appendix A, since 2011, is approximately 1.8 mils/year. This observed corrosion rate is reasonably close to the target rate for mass loss coupons in GM 9540P of approximately 1.4 mils/year. According to Weatherbase,²¹ over the past 28 years the average temperature has been 71 °F and average rainfall is 44.8 inches. The observed average temperature during the exposure period was 74.8 °F and the average observed rainfall was 25.3 inches.

2.8 Performance Objectives

The performance objectives and the success criteria are defined in Table 7. The success criteria for all of the pretreatments tested were based on several factors. The first source considered was TT-C-490 Revision F. For products to be qualified for use, they must meet the requirements for inclusion in the specifications qualified product database. These requirements have been integrated in the performance objectives in Table 7. Additional requirements that are not reflected in TT-C-490F were determined using the relative performance of the alternatives to the standard DOD-P-15328 wash primer.

Table 7 Performance objectives with success criteria

Performance Objective	Data Requirements	Success Criteria
Adhesion Test	ASTM D3359 Dry Adhesion ASTM- D3359 Wet Adhesion/Fed Std 141 6301.3	Adhesion rating (all substrates) ≥ 4 ; Adhesion rating (all substrates) ≥ 4 ;
Coating Hardness	Pencil Hardness ASTM D3363	Film softening shall not exceed two pencil hardness difference from baseline
Chip Resistance	SAE-J400	All substrates: Rating for number of chips ≥ 5 Rating for size of chips $\geq B$
Corrosion Resistance	ASTM B117 Salt Fog ASTM D1654	After 336/1000* hours of exposure: Steel substrate rating ≥ 6 scribed, ≥ 7 BIF** Aluminum substrate rating ≥ 8 scribed, ≥ 7 BIF** Multimetal combined rating ≥ 6 scribed, ≥ 7 BIF**
	GM 9540P Cyclic Corrosion ASTM D1654	After 40 cycles of exposure***: Steel substrate rating ≥ 6 scribed, ≥ 7 BIF** Aluminum substrate rating ≥ 8 scribed, ≥ 7 BIF** Multimetal combined rating ≥ 6 scribed, ≥ 7 BIF**
Flash Rust inhibition	modified ASTM D1735	No flash rust after 24 hours of exposure to ambient temperature and 90% relative humidity
Outdoor Exposure	Cape Canaveral ASTM D1654 ASTM G50	Two years of exposure: Steel substrate rating \geq baseline Aluminum substrate rating \geq baseline Multimetal combined ratings \geq baseline
Toxicity Clearance	Toxicity clearances and full disclosure from Public Health Command	Approved by processing facility
Processing time	TT-C-490F	Equivalent or less than existing process
Field Testing	TT-C-490F	After 2 year outdoor exposure rating \geq current technology
Qualitative Performance Objectives		
Ease of use	Feedback from field technician on usability of technology and time required during demonstration	No formal operator training required. Training from supplier technical representative.

* 336 hours for MIL-DTL-53022 Type II, 1000 hours for MIL-DTL-53030 Type IV

** Blisters in Field (BIF), no single blister in excess of 3mm

*** No cyclic requirement for MIL-DTL-53022 Type II

3. Results and Discussion

3.1 Wet Tape Adhesion

All adhesion testing was done on mill-finish panels. That is, there was no abrasive blasting or appreciable profile on any of the 4 substrates tested (Al, CRS, galvanized steel, and stainless steel). Table 8 lists the ratings for the wet tape adhesion tests for all candidates and baselines along with the color coding for met/not met (green = met and red = not met) of the success criteria listed in Table 8 for wet tape adhesion. The success criterion for wet tape adhesion is a rating greater than or equal to 4. The baseline DOD-P-15328 wash primer

performed well, meeting the success criterion with both MIL-DTL-53022 and MIL-DTL-53030 primers on all substrates. The Oxsilan 9810/2, and Ecosil (ECO 5-1) performed better than the wash primer—achieving ratings of 5 in many cases. PPG 11-TGL-07-Z (Zircobond), Bonderite 7400 and SurTec 650 showed similar performance as the wash primer and met the success criterion in most cases, galvanized surfaces being the exception. The SurTec 650 clearly did not perform well on galvanized surfaces, while the Bonderite 7400 failed with only the MIL-DTL-53030 primer.

Picklex, AC-10, and the NCP nonchromated wash primer had difficulty meeting the wet tape adhesion criterion across all substrates. The NCP nonchromate wash primer showed very poor wet tape adhesion, failing to meet the success criterion with any substrate/primer combination. On average, Ecosil outperformed the wash primer and all other candidates in wet tape adhesion with all substrate/primer combinations.

Table 8 Wet tape adhesion ratings (ASTM D3359 Method A)

		Wet Adhesion			
		Al	CRS	Galv	SS
Picklex 20	53022	0	2	3	0
	53030	3	3	3	4
SurTec 650	53022	4	4	2	4
	53030	4	5	2	5
Aero-Green AC-10	53022	3	4	5	0
	53030	4	3	3	4
PPG 11-TGL-07-Z	53022	4	4	4	4
	53030	4	4	5	4
Oxsilan 9810/2	53022	4	4	4	4
	53030	5	5	5	4
Bonderite 7400	53022	4	5	5	4
	53030	5	5	0	5
Ecosil Eco 5-1	53022	5	5	5	5
	53030	5	5	5	4
NCP WP N-8237-2.5 A/B	53022	0	1	0	1
	53030	0	3	3	2
DOD-P-15328 WP	53022	4	4	4	4
	53030	4	4	4	4

3.2 Dry Tape Adhesion

As in the wet tape adhesion testing, dry tape adhesion (or cross hatch) testing was done on mill-finish versions on the same 4 substrates as above. No appreciable surface profile was given to any of the substrates.

Table 9 lists the ratings for the dry tape adhesion tests for all candidates and baselines along with the color coding for met/not met (green = met and red = not met) of the success criteria listed in Table 9 for dry tape adhesion. The success criterion for dry tape adhesion is a rating greater than or equal to 4. As with the wet tape adhesion, the baseline DOD-P-15328 wash primer met the success criterion with MIL-DTL-53022 and MIL-DTL-53030 primers on all substrates.

Table 9 Dry tape adhesion ratings (ASTM D3359 Method B)

		Cross Hatch Adhesion			
		Al	CRS	Galv	SS
Picklex 20	53022	0	0	4	4
	53030	0	1	4	4
SurTec 650	53022	2	4	0	4
	53030	4	3	2	5
Aero-Green AC-10	53022	3	3	5	0
	53030	3	2	2	4
PPG 11-TGL-07-Z	53022	4	3	5	4
	53030	4	4	3	5
Oxsilan 9810/2	53022	5	4	5	5
	53030	5	3	5	4
Bonderite 7400	53022	4	4	4	4
	53030	5	5	2	5
Ecosil Eco 5-1	53022	5	5	5	5
	53030	5	5	4	5
NCP WP N-8237-2.5 A/B	53022	0	0	0	2
	53030	0	4	4	4
DOD-P-15328 WP	53022	4	4	4	4
	53030	4	4	4	4

A similar trend is seen in dry tape adhesion testing as was seen in wet tape adhesion testing as to how each product performs on each substrate with each primer. Oxsilan 9810/2, Ecosil, and Bonderite 7400 performed similar to or better than wash primer in most situations. PPG 11-TGL-07-Z (Zircobond) and SurTec 650 performed less favorably than they had in wet adhesion testing. Overall, the SurTec 650, Picklex, AC-10, and NCP nonchromated wash primer performed at a substantially lower level than baseline wash primer. The difference in performance of the primers was much less distinct in dry adhesion testing. There was also not much difference in the adhesive performance of the wash primer alternatives across each substrate save for stainless steel, which was almost an entire rating point higher than the other 3 substrates when averaged. Overall, Ecosil again outperformed the wash primer and all other candidates in dry tape adhesion with all substrate/primer combinations.

3.3 Pencil Hardness

There was little variation in the pencil hardnesses of the coatings across each substrate/primer/pretreatment combination. The success criteria in Table 7 states that film softening shall not exceed two pencil hardness difference from baseline. The DOD-P-15328 has a baseline pencil hardness rating for each primer. For MIL-DTL-53022, the baseline hardness is “B” and for MIL-DTL-53030 the baseline hardness is HB. All of the combinations in Table 10 met the success criteria for pencil hardness, indicating that the pretreatments had no significant affect on the hardness of the coating system. The hardest rating was an “F” and was on Al and stainless steel panels treated with Oxsilan and primed with MIL-DTL-53022. The lowest reported pencil hardness was 2B, and was seen on many of the NCP wash primer combinations, but was still within the two hardness rating requirement. The 2B grades seen on the NCP wash primer reflects the issues with adhesion displayed in the previous two adhesion tests. The lack of significant variance in the pencil hardness across each specimen shows that most of the pretreatments tested had no effect on the chemistries of the primers and topcoat.

Table 10 Pencil hardness ratings (ASTM D3363)

		Pencil Hardness			
		Al	CRS	Galv	SS
Picklex 20	53022	HB	B	B	B
	53030	HB	HB	B	HB
SurTec 650	53022	HB	HB	HB	HB
	53030	B	HB	HB	2B
Aero-Green AC-10	53022	2B	B	B	B
	53030	B	B	B	B
PPG 11-TGL-07-Z	53022	HB	HB	HB	B
	53030	HB	HB	B	HB
Oxsilan 9810/2	53022	F	2B	HB	F
	53030	HB	B	B	HB
Bonderite 7400	53022	HB	HB	B	HB
	53030	B	B	B	HB
Ecosil Eco 5-1	53022	HB	HB	HB	B
	53030	B	B	HB	HB
NCP WP N-8237-2.5 A/B	53022	2B	2B	HB	B
	53030	2B	2B	2B	2B
DOD-P-15328 WP	53022	B	B	B	HB
	53030	B	HB	HB	HB

3.4 Chip Resistance Testing

Table 11 shows the ratings for SAE J400 chip resistance. The pretreatments varied across each of the substrates with stainless steel having the lowest ratings. The Bonderite 7400, Ecosil ECO 5-1, the baseline wash primer, Oxsilan 9810/2 and PPG 11-TGL-07-Z performed well regardless of substrate. The Picklex product showed poor resistance to chipping and was the worst of all the products, unable to meet the success criterion on any substrate. The chip resistance test results appear to be congruent with the results seen in the previous 3 adhesion tests. In general, those that provided the best adhesion also were most resistant to chipping.

Table 11 Chip resistance ratings per SAE J400

Pretreatment	Primer	Chip Resistance, SAE J400 Ratings			
		Al	CRS	Galv	SS
Picklex	53022	1D	8D	9C	5D
	53030	2D	5D	7C	5D
SurTec 650	53022	7A	9D	8D	6C
	53030	6A	6A	6A	6B
AC-10	53022	6C	9A	9A	5D
	53030	4D	8B	8C	2C
PPG 11-TGL-07-Z	53022	7B	7A	7A	7C
	53030	7B	6A	6A	6C
Oxsilan 9810/2	53022	7A	7A	8A	7B
	53030	7B	6A	6A	6C
Bonderite 7400	53022	7A	8A	6B	6C
	53030	7B	6A	7A	7B
Ecosil ECO 5-1	53022	8A	8A	9B	7B
	53030	7A	7A	7A	5C
NCP WP	53022	5C	9B	8A	7D
	53030	6D	7C	6B	5D
DOD-P-15328	53022	7C	7A	7A	5B
	53030	6B	6A	6A	5B

Figures 9–12 show the results of the SAE J400 test panels after testing. Although the ratings quantitatively determine whether the pretreatments met or did not meet the success criteria, the photographs below show the performance qualitatively. Ecosil 5-1 and the Bonderite 7400 were 2 of the most resistant to chipping along with the Oxsilan 9810/2 (not shown). These alternatives compare very well to the baseline wash primer in Fig. 11. In fact, qualitatively, one could suggest the alternatives were better at resisting chipping. In contrast, Fig. 12 shows the poor performance of the Picklex with all primers and substrates. The chip resistance is particularly important for ground vehicles to prevent damage to the CARC system.



Fig. 9 Bonderite 7400 on (from left to right) Al, CRS, galvanized, and stainless steel (53030 top, 53022 bottom)

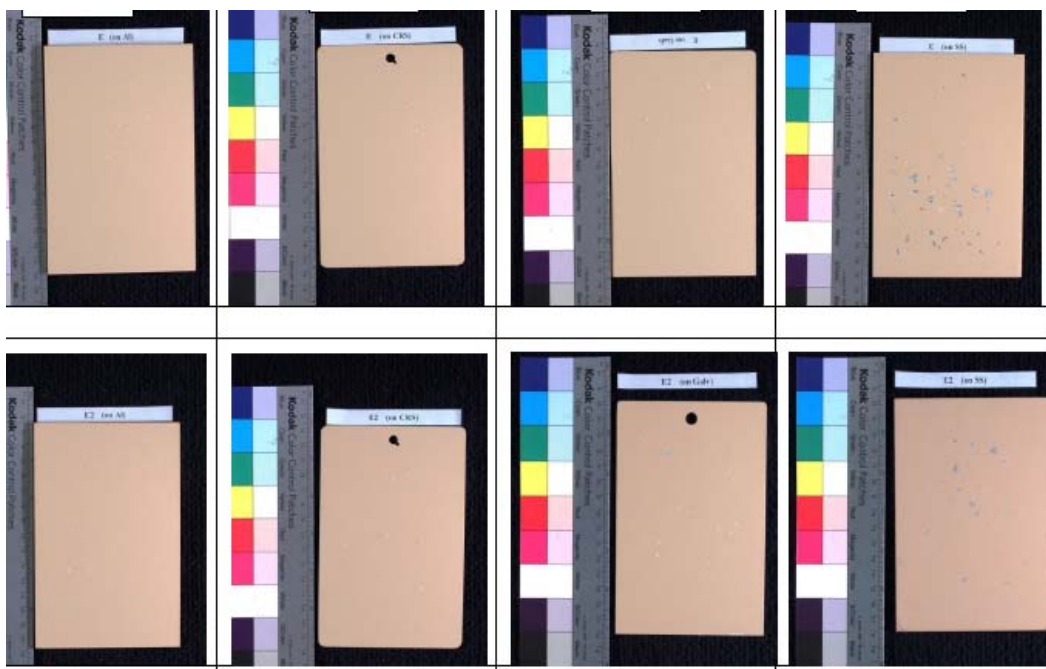


Fig. 10 Ecosil 5-1 on (from left to right) Al, CRS, galvanized, and stainless steel (53030 top, 53022 bottom)



Fig. 11 DOD-P-15328 wash primer on (from left to right) Al, CRS, galvanized, and stainless steel (53030 top, 53022 bottom)

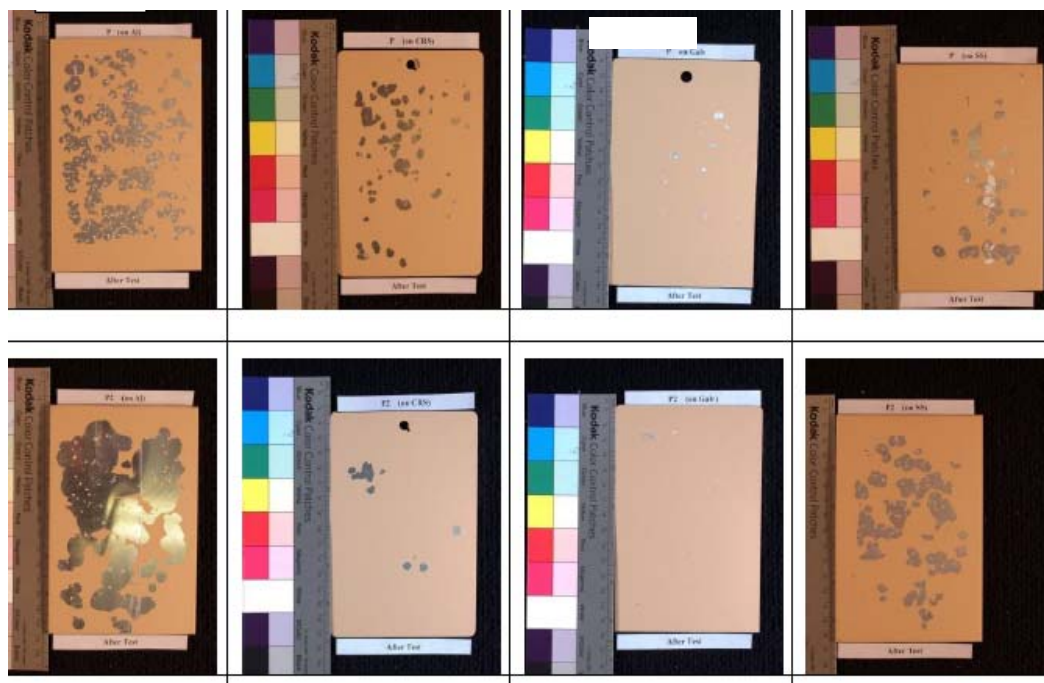


Fig. 12 Picklex on (from left to right) Al, CRS, galvanized, and stainless steel (53030 top, 53022 bottom)

3.5 Neutral Salt Fog Corrosion (ASTM B117)

The success criteria for testing IAW ASTM B117 salt fog was derived from TT-C-490F and is listed in Table 7. MIL-DTL-53022 and MIL-DTL-53030 primers are evaluated after 336 h and 1,000 h, respectively. To meet the performance objectives, steel test panels must achieve a creepage from scribe rating greater than or equal to 6 and blisters in field must be greater than or equal to 7. For Al panels to meet performance objectives, the creepage from scribe rating must be greater than or equal to 8 and blisters in field greater than or equal to 7. The multimetal specimens use a rating for the Al section combined with the steel section and the ratings shall be greater than or equal to 6 scribed, with greater than or equal to 7 blisters in field. In all cases, no single blister in field shall exceed 3-mm width. Unfortunately, the project has had some delays caused by accelerated corrosion chamber downtime. Many of the test panels have not yet been exposed through the required 1,000 h. The intent is to make our best assessment through 672 h and amend if necessary after the 1,000 h has been reached.

CRS panels were primed with MIL-DTL-53030 and MIL-DTL-53022 were tested through 672 h in salt fog testing. The ASTM D1654 ratings for all pretreatments and baseline on CRS can be seen in Table 12. All pretreatments met the success criteria of greater than or equal to 6 after 336 h with MIL-DTL-53022. When evaluated thus far through 672 h for the MIL-DTL-53030, the only pretreatments that are meeting the success criteria are Bonderite 7400, Ecosil 5-1, baseline wash primer, and the NCP wash primer. There was no blistering in the unscribed field on any panel through 672 h, though staining from the edges and the scribe became prevalent over time. These stains are not accounted for during the rating process.

Table 12 **ASTM D1654 ratings for ASTM B117 neutral salt fog testing on CRS**

Pretreatment		Exposure Time (hours)			
		Creep from Scribe		Blisters in Field	
		336	672	336	672
Picklex 20	53022	7.6	6.4	10	10
	53030	4.4	3.6	10	10
SurTec 650	53022	8.2	6.6	10	10
	53030	8	5.8	10	10
Aero-Green AC-10	53022	6.6	5.6	10	10
	53030	6	5.6	10	10
PPG 11-TGL-07-Z	53022	8.6	4.4	10	10
	53030	5.2	3.6	10	10
Oxilan 9810/2	53022	7.6	5.6	10	10
	53030	6.2	5.4	10	10
Bonderite 7400	53022	8.4	6.6	10	10
	53030	8.4	7	10	10
Ecosil Eco 5-1	53022	7.8	6.4	10	10
	53030	8	6.2	10	10
NCP WP N-8237-2.5 A/B	53022	7.6	7.2	10	10
	53030	7	6.2	10	10
DOD-P-15328 WP	53022	9	7.2	10	10
	53030	8.4	6.6	10	10

Al panels were run out to 672 h of exposure in ASTM B117 testing. The results obtained can be seen in Table 13 below. At 672 h, only the Bonderite 7400 and DOD-P-15328 wash primer is meeting the success criteria of greater than or equal to 8, and that is only with the MIL-DTL-53030 CARC primer. No pretreatments on Al with MIL-DTL-53022 are meeting the success criteria for creep-from-scribe after 672 h. The Picklex treated panels primed with 53022 had completely delaminated after 72 h and the AC-10 treated panels primed with 53022 were completely delaminated after 168 h. The Bonderite 7400 performed slightly better than that of the control wash primer. Some were very close to the required greater than or equal to 8 rating. Although the SurTec 650 (trivalent chrome Al pretreatment) did not perform as well on steel substrates, it had a consistent 7.8 with both primer types. Bonderite 7400 and NCP nonchromate wash primer rated a 7.4 and 7.6, respectively. The ratings for all Al samples are expected to be lower once the required 1000 h of salt fog exposure is reached. The reason for the low ratings on Al is likely because Al should be deoxidized prior to pretreatment. The native oxide of the Al hinders proper reaction of the conversion coatings.

Table 13 **ASTM D1654 ratings for ASTM B117 neutral salt fog testing on Al**

		Exposure Time (hours)			
		Creep from Scribe		Blisters in Field	
Pretreatment		336	672	336	672
Picklex 20	53022	0	0	10	10
	53030	4.6	2.8	10	10
SurTec 650	53022	7.8	7.8	10	10
	53030	8.4	7.8	10	10
Aero-Green AC-10	53022	0	0	10	10
	53030	3.2	2.6	10	10
PPG 11-TGL-07-Z	53022	6.4	4.6	10	10
	53030	6	4	10	10
Oxsilan 9810/2	53022	7	6.4	10	10
	53030	2.4	1.2	10	10
Bonderite 7400	53022	7.8	7.4	10	10
	53030	9	9	10	10
Ecosil Eco 5-1	53022	3.8	3.8	10	10
	53030	3.6	2	10	10
NCP WP N-8237-2.5 A/B	53022	7.6	7.6	10	10
	53030	6.4	6.4	10	10
DOD-P-15328 WP	53022	7.2	7.2	10	10
	53030	9	9	10	10

Figures 13–15 are photographs of MIL-DTL-53022 panel sets through 672 h. At 672 h, both the ratings and photographs show that the Bonderite 7400 and compares very well with the baseline wash primer on both steel and Al. The Picklex shown in Fig. 15, rated 0.0, along with AC-10 showed early catastrophic failures at only 72 h.



Fig. 13 Bonderite 7400 rated a 7.4 on Al panels with MIL-DTL-53022 at 672 h



Fig. 14 DOD-P-15328 rated a 7.2 on Al panels with MIL-DTL-53022 at 672 h



Fig. 15 Picklex rated a 0.0 on Al panels with MIL-DTL-53022 at only 72 h

Multimetal panels were exposed through 672 h of B117 testing. The success criteria for these samples takes into consideration all of the exposed surface area for blisters in the field. The ratings for the multimetal specimens can be seen in Table 14. Similar to what was discussed earlier, coating adhesion on the extruded Al “L” bracket was challenging. Additionally, the galvanized fastener and surrounding area tended to show more corrosion than that of the stainless steel fastener. The hydroxide film exuded by the galvanized fastener tended to build up over time in the Al “L” bracket and was not easily removed by the fog or rinsing during inspections. Staining around the U-weld was common on all specimens. As can be seen in Table 12, most of the pretreatments and baseline met the success criteria including DTM, which has no pretreatment. Interestingly, the DTM samples performed very similarly to the wash primer samples. The Bonderite 7400 performed consistently across all substrates and primers easily meeting all of the success criteria. However, as previously mentioned, the ratings for all MIL-DTL-53030 samples are expected to be lower once 1000 h of exposure is achieved.

Table 14 ASTM D1654 ratings for ASTM B117 salt fog testing on multimetal panels

		Exposure Time (hours)			
		Creep from Scribe		Blisters in Field	
Pretreatment		336	672	336	672
Picklex 20	53022	6.8	5.4	7	7
	53030	8.6	8.6	9.4	7
SurTec 650	53022	7	6.2	7.6	7.2
	53030	8.2	7.2	7.8	6.2
Aero-Green AC-10	53022	8.2	7.6	7	7
	53030	9	7.2	9.4	8.8
PPG 11-TGL-07-Z	53022	7.2	6	9	7
	53030	7.2	6.6	8.8	8
Oxsilan 9810/2	53022	7.4	6.2	8	6.6
	53030	7.4	7.4	8.4	7.4
Bonderite 7400	53022	9	8.2	9.4	8
	53030	9	8.8	8.6	8.6
Ecosil Eco 5-1	53022	7.8	6.8	8	7.6
	53030	8.6	7	7.2	6.8
NCP WP N-8237-2.5 A/B	53022	5.4	4.8	8.6	6.6
	53030	8.4	7.8	10	9.4
DOD-P-15328 WP	53022	7	6.4	8	6.8
	53030	8.8	7.8	10	10
Direct to Metal	53022	9	7.4	7.6	6
	53030	8.8	7.8	10	10

An example of the performance of Bonderite 7400 versus the baseline wash primer can be seen in Figs. 16 and 17. Although the success criteria were met after 336 h of ASTM B117 exposure, these photographs were taken after 672 h. Although the Bonderite 7400 samples show significantly more staining and edge corrosion than the wash primer, there is less undercutting at the scribe on average than the wash primer. The chromate in the wash primer is clearly inhibiting the corrosion of the steel and preventing the staining seen in the other alternatives. The coating adhesion provided by the Bonderite 7400 resists the undercutting better than the wash primer.



Fig. 16 Bonderite 7400 on multimetal panels with MIL-DTL-53022 after 672 h



Fig. 17 DOD-P-15328 on multimetal panels with MIL-DTL-53022 after 672 h

3.6 Accelerated Cyclic Corrosion (GM 9540P)

CRS panels were exposed to 80 cycles of GM 9540P cyclic corrosion testing; however, the success criteria is evaluated after 40 cycles of exposure. For steel substrate a rating greater than or equal to 6 creep-from-scribe, and the blisters in field rating greater than or equal to 7. For Al a rating of greater than or equal to 8 creep-from-scribe, and greater than or equal to 7 for blisters in field. The multimetal samples require a rating greater than or equal to 6 scribed, and combined ratings greater than or equal to 7 blisters in the field to include Al and U-weld areas.

The ASTM D1654 ratings for all pretreatments on CRS are presented in Table 15. At 40 cycles, only the baseline wash primer, Bonderite 7400, and Ecosil 5-1 met the success criteria on panels primed with either MIL-DTL-53022 or MIL-DTL-53030. Bonderite maintained the highest average rating on CRS of the 3 pretreatments that passed. AC-10 was relatively close to meeting the requirements, with 3 of 5 panels passing with ratings of 6. The other 2 panels rated 5 and kept the average below the threshold acceptable rating of 6.

While all of the pretreatments met the blisters in field requirement, only the NCP nonchromate wash primer, Oxsilan 9810/2, and Picklex-treated panels showed any blistering through the entire 80 h.

After the 80 cycles were completed, all panels were scraped with a 2-inch-flat-blade putty knife after rating to unveil any previously unseen corrosion or delamination issues between the coating and the substrate. After scraping, only a small amount of hidden corrosion on an AC-10 treated panel was revealed. It is important to note that only the Bonderite 7400 met the success criteria with MIL-DTL-53030 and nearly met with MIL-DTL-53022 after 80 cycles. In addition to the Bonderite 7400, the Ecosil 5-1 also performed well and met the success criteria through 60 cycles.

Table 15 **ASTM D1654 ratings for GM 9540P cyclic corrosion testing on CRS**

		Exposure Time (cycles)							
		Creep from Scribe				Blisters in Field			
Pretreatment		20	40	60	80	20	40	60	80
Picklex 20	53022	6	4	3	2.2	10	10	10	8
	53030	6.25	5	5	4	10	10	10	10
SurTec 650	53022	6.2	2.6	1.4	0	10	10	10	8
	53030	6.6	4.2	3.4	3.4	10	10	10	10
Aero-Green AC-10	53022	7.4	5.8	5.4	4.8	10	10	10	10
	53030	7.6	5.6	5.4	5.2	10	10	10	10
PPG 11-TGL-07-Z	53022	5.8	3.2	1.8	0.4	10	10	10	10
	53030	6	3.5	3.5	2.5	10	10	10	10
Oxsilan 9810/2	53022	5.4	1.6	0.4	0.2	10	10	8	2
	53030	6.6	3.8	3	2	10	10	10	10
Bonderite 7400	53022	9	6.25	5.75	5.5	10	10	10	10
	53030	9	6.75	6.75	6	10	10	10	10
Ecosil Eco 5-1	53022	9	6.25	6	5.25	10	10	10	10
	53030	8.75	6	6	5.25	10	10	10	10
NCP WP N-8237-2.5 A/B	53022	5.6	3	2	0.8	10	10	10	10
	53030	6	3	2.4	1.4	10	8	8	8
DOD-P-15328 WP	53022	6.8	6.4	5.8	5	10	10	10	10
	53030	8.25	6.5	6.5	5.5	10	10	10	10

The following is presented to illustrate the performance of the 2 alternatives that met the success criteria for GM 9540P (Figs. 18–23) relative to the baseline wash primer in Figs. 18–23. After the panels were exposed to 80 cycles and subsequently scraped, it is difficult to discern any difference in performance.

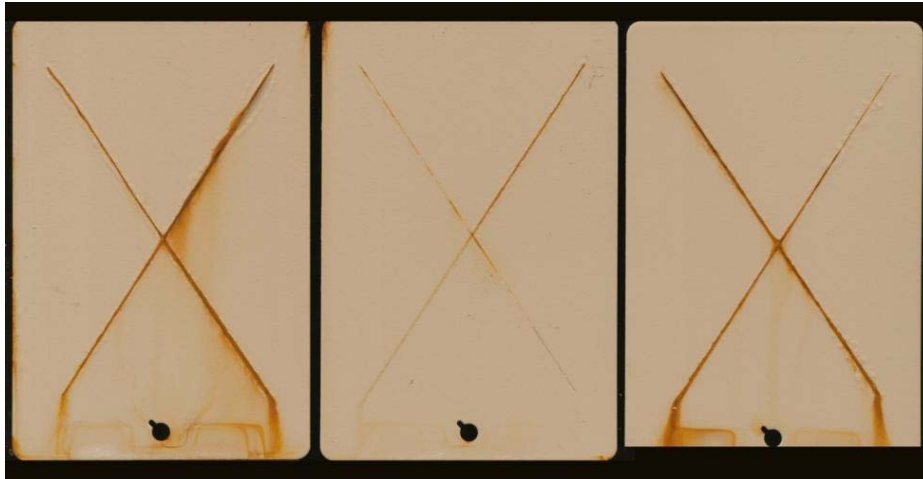


Fig. 18 Bonderite 7400 on CRS primed with MIL-DTL-53022 after 80 cycles



Fig. 19 Bonderite 7400 on CRS primed with MIL-DTL-53030 after 80 cycles



Fig. 20 Ecosil 5-1 on CRS primed with MIL-DTL-53022 after 80 cycles



Fig. 21 Ecosil 5-1 on CRS primed with MIL-DTL-53030 after 80 cycles



Fig. 22 DOD-P-15328 on CRS primed with MIL-DTL-53022 after 80 cycles

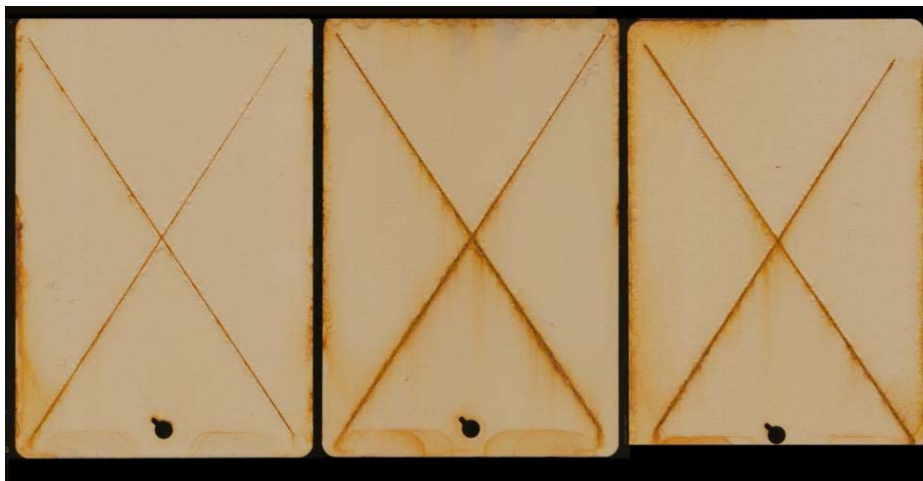


Fig. 23 DOD-P-15328 on CRS primed with MIL-DTL-53022 after 80 cycles

Although success was established at 40 cycles, the Al test panels were also run out to 80 cycles to determine longer term effectiveness. To meet the success criteria, the Al test panels must be rated 8.0 or higher at 40 cycles. All of the ratings for ASTM D1654 ratings for GM 9540P on Al are presented in Table 16. As discussed previously, it is more challenging for some of these alternative pretreatments to be effective on Al because it is typically deoxidized as part of the pretreatment/conversion coating process. The SurTec 650 was expected to meet the success criteria because it was developed as a conversion coating for Al. In addition to SurTec 650, Oxsilan 9810/2 and PPG 11-TGL-07 Z also performed well. Each of these products is a zirconium-modified pretreatment. Although the Bonderite was unable meet the success criteria with the MIL-DTL-53022 primer, it was only 0.2 below the pass threshold of an average rating of 8.

Table 16 **ASTM D1654 ratings for GM 9540P cyclic corrosion testing on Al**

Pretreatment		Exposure Time (cycles)							
		Creep from Scribe				Blisters in Field			
		20	40	60	80	20	40	60	80
Picklex 20	53022	1.8	0	0	0	10	6	0	0
	53030	6.2	5.6	2.4	0	10	10	10	8
SurTec 650	53022	10	9	8.2	8.2	10	10	10	10
	53030	10	9	8.8	8.8	10	10	10	10
Aero-Green AC-10	53022	5	3	0	0	10	10	10	0
	53030	4.2	2.6	0.6	0	10	6	6	2
PPG 11-TGL-07-Z	53022	10	9	8	7.4	10	10	10	10
	53030	10	9	9	8.8	10	10	10	10
Oxsilan 9810/2	53022	9.6	8.6	8.6	8.4	10	10	10	10
	53030	10	9	8.2	8	10	10	10	10
Bonderite 7400	53022	8.2	7.8	7	6.8	10	10	10	10
	53030	9.8	9	8.8	8.6	10	10	10	10
Ecosil Eco 5-1	53022	9.2	8.4	6	5.8	10	10	10	10
	53030	10	9	7.2	5.2	10	10	10	10
NCP WP N-8237-2.5 A/B	53022	5.8	4	2.2	1.8	10	8	8	4
	53030	10	8.4	0	0	10	10	10	10
DOD-P-15328 WP	53022	10	8.8	8	7.8	10	10	10	10
	53030	10	9	9	8.8	10	10	10	10

It is clear that Picklex and AC-10 did not perform well on Al. Both did not achieve more than 6.2 after 20 cycles. In addition, the NCP wash primer failed catastrophically, delaminating between the 40 and 60 cycle inspections with both primers.

The best performing pretreatments on Al with either primer in cyclic testing considering success criteria were: the baseline DOD-P-15328 wash primer, SurTec 650, PPG 11-TGL-07 Z Oxsilan 9810/2, Ecosil 5-1, and Bonderite 7400. Considering each product's performance on steel substrates to this point, Bonderite 7400 and Ecosil 5-1 have shown promise as viable alternatives to the baseline wash primer. The images in Figs. 24–29 show little difference in the visual appearance between the baseline wash primer Bonderite 7400 and the Ecosil 5-1 after 80 cycles. Even with the lower creep-from-scribe ratings both appear suitable for use on Al.

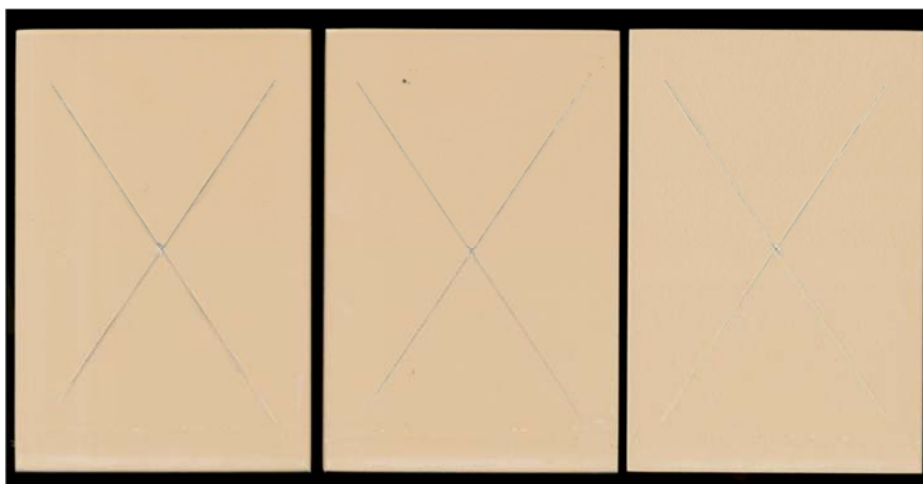


Fig. 24 Bonderite 7400 on Al primed with MIL-DTL-53022 after 80 cycles



Fig. 25 Bonderite 7400 on Al primed with MIL-DTL-53030 after 80 cycles

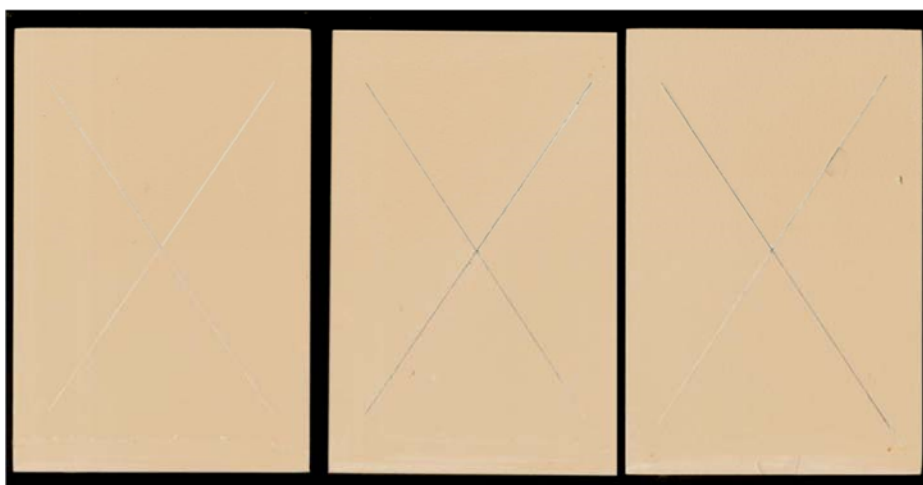


Fig. 26 Ecosil 5-1 on Al primed with MIL-DTL-53022 after 80 cycles

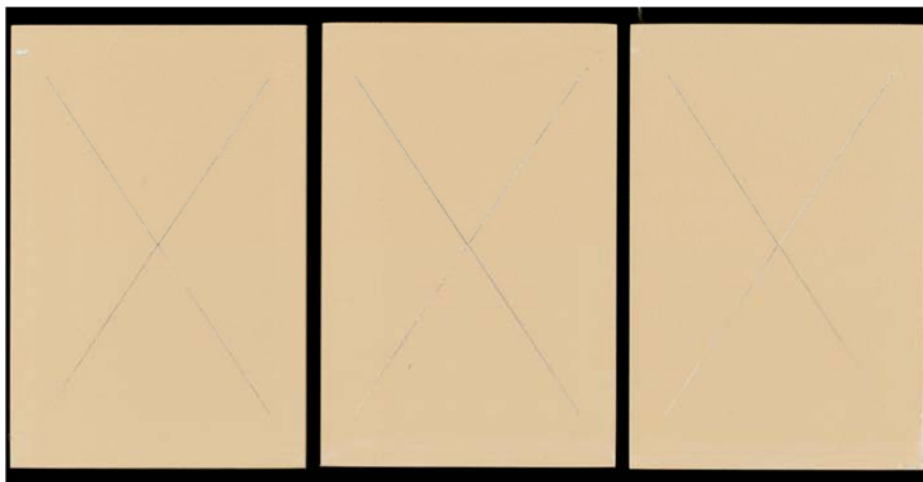


Fig. 27 Ecosil 5-1 on Al primed with MIL-DTL-53030 after 80 cycles

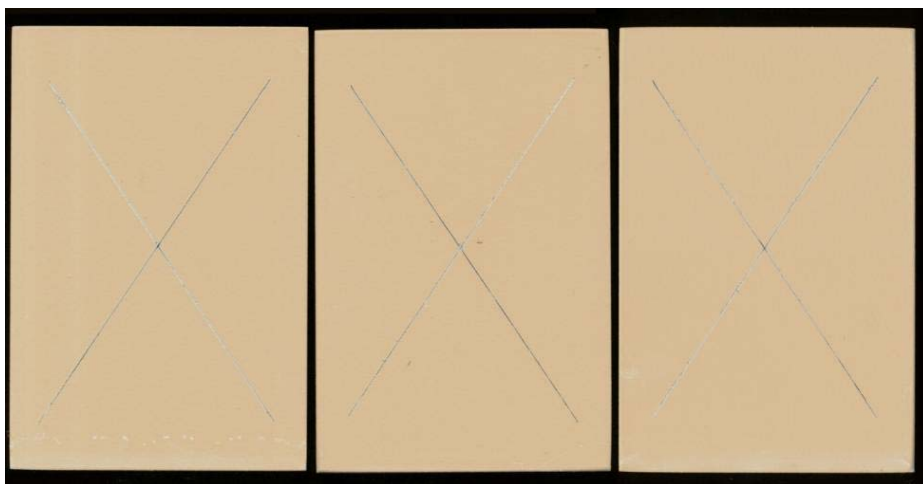


Fig. 28 DOD-P-15328 on Al primed with MIL-DTL-53022 after 80 cycles

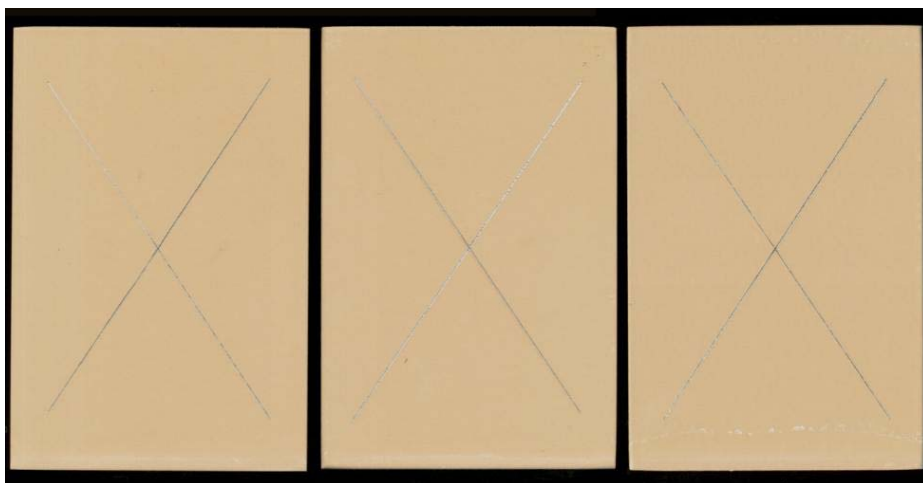


Fig. 29 DOD-P-15328 on Al primed with MIL-DTL-53030 after 80 cycles

Because of the more complex geometry and dissimilar metals, it was expected that this sample would be more challenging for all pretreatments including the baseline wash primer. This expectation is verified with the results presented in Table 17. The multimetal samples require a rating greater than or equal to 6 scribed, and combined ratings of greater than or equal to 7 blisters in the field to include Al and U-weld areas after 40 cycles. Only 2 pretreatments were able to meet the success criteria with both primers at 40 cycles: Bonderite 7400 and Oxsilan 9810/2. In fact, the only alternatives that met the success criteria with the MIL-DTL-53022 primer were the Bonderite 7400 and the Oxsilan 9810/2. Beyond that, no pretreatment was completely effective with both MIL-DTL-53022 and MIL-DTL-53030. Similarly, there was a significant amount of variation in ratings for blisters in field. The Bonderite 7400 and the Oxsilan 9810/2 met the success criteria as did the baseline. The MIL-DTL-53030 Type IV clearly provides better corrosion protection than the MIL-DTL-53022 Type II. Many of the alternatives performed as well as or better than the baseline wash primer when primed with the MIL-DTL-53030 primer. The only alternative pretreatment system that failed at 40 cycles with the MIL-DTL-53030 primer was the NCP nonchromate wash primer. The NCP nonchromate wash primer coupons failed catastrophically after just a few cycles. A photograph was taken of these panels after being removed from the test chamber and can be seen in Fig. 30.

Table 17 **ASTM D1654 ratings for GM9540P testing on “multimetal” specimens**

Pretreatment		Exposure Time (cycles)							
		Creep from Scribe				Blisters in Field			
		20	40	60	80	20	40	60	80
Picklex 20	53022	3.2	0.8	0.4	0	4.2	3.2	1	0
	53030	8	7.8	7.4	6.8	8.6	8.2	7.6	7
SurTec 650	53022	4.8	3.6	2.6	1.6	6.6	6.2	4.2	3
	53030	7.2	6	6	6	9.2	8	6.8	6.4
Aero-Green AC-10	53022	6.2	5.4	5	4.8	8.2	6.8	5.8	5.8
	53030	8	6.4	6.2	6	10	10	8.8	8.8
PPG 11-TGL-07-Z	53022	6.4	5.4	4.8	4.8	9.4	7.8	6.4	6.2
	53030	8	6.4	6.4	6.4	10	8.6	8	7.2
Oxsilan 9810/2	53022	7	6.4	5	5	8.4	7.4	6.6	6.6
	53030	8.8	8	8	7.6	10	10	8.2	8.2
Bonderite 7400	53022	7.6	6.8	5.2	5	8.4	7.6	6.6	6.4
	53030	8.2	7	7	6.8	10	9.6	9.2	8.8
Ecosil Eco 5-1	53022	5.2	4.2	3.2	2.2	7	7	6	3.6
	53030	8.4	7.2	7.2	7.2	9.8	9.4	8.6	8.6
NCP WP N-8237-2.5 A/B	53022	0	0	0	0	0	0	0	0
	53030	7.4	5.2	4.8	4	9.2	8.4	7	5.8
DOD-P-15328 WP	53022	5.6	4.2	3	2	9.8	8.8	8	5.4
	53030	8.6	7.4	7.2	7.2	10	10	9.8	9.8
Direct to Metal	53022	6.2	4.8	3.6	3	9.2	8	5.6	5
	53030	8.4	6.6	6.6	6.4	9.2	6.2	5.6	5.6



Fig. 30 NCP nonchromate wash primer multimetal panels delaminating after 3 cycles

Figures 31–36 show the multimetal galvanic test specimen after 80 cycles. One can clearly see the superior corrosion protection provided by the MIL-DTL-53030 Type IV versus the MIL-DTL-53022 Type II primer. Examining only the MIL-DTL-53022 Type II specimens, it is evident that there is less blistering along the scribes of the Bonderite 7400 specimens than on the baseline wash primer or the Ecosil 5-1 specimens. However, there is noticeably more delamination at the Al “L” bracket for Bonderite 7400 and Ecosil 5-1 than is seen on the baseline wash primer. Note that the MIL-DTL-53030 shows no delamination at the Al angle bracket.

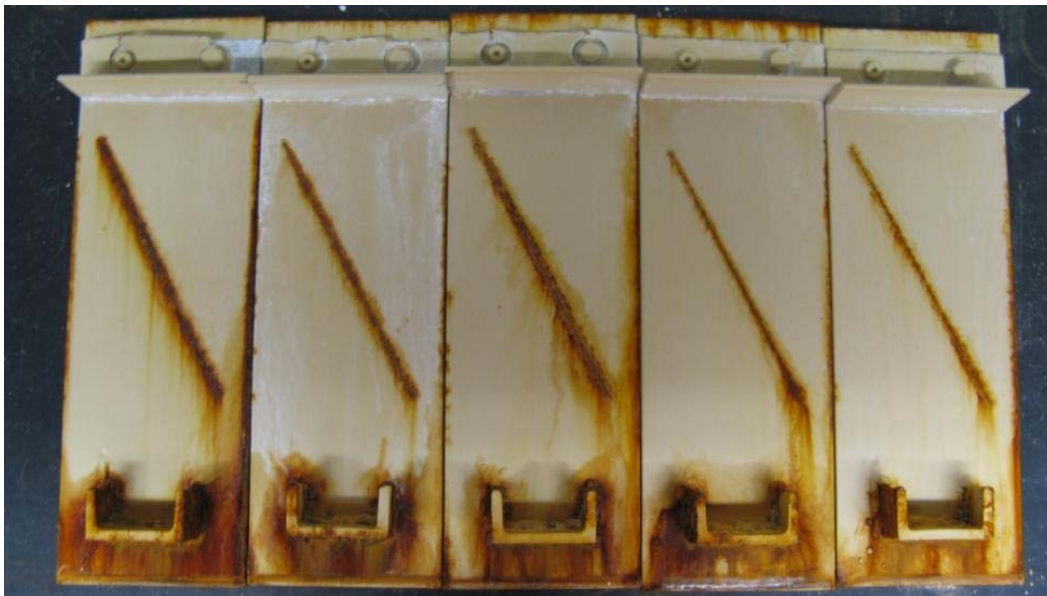


Fig. 31 Bonderite 7400 on multimetal panels primed with MIL-DTL-53022 after 80 cycles



Fig. 32 Bonderite 7400 on multimetal panels primed with MIL-DTL-53030 after 80 cycles



Fig. 33 Ecosil 5-1 on multimetal panels primed with MIL-DTL-53022 after 80 cycles



Fig. 34 Ecosil 5-1 on multimetal panels primed with MIL-DTL-53030 after 80 cycles

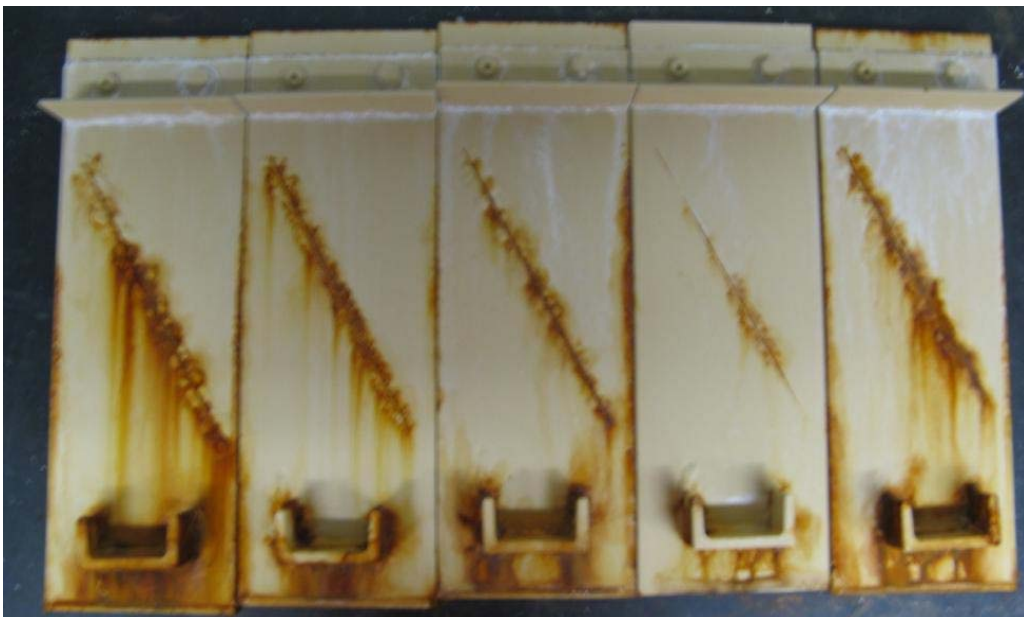


Fig. 35 DOD-P-15328 on multimetal panels primed with MIL-DTL-53022 after 80 cycles



Fig. 36 DOD-P-15328 on multimetal panels primed with MIL-DTL-53030 after 80 cycles

3.7 Outdoor Exposure

Outdoor exposure test coupons of Al, CRS, as well as multimetal assemblies were initiated at CCAFS on September 26, 2013. The success criteria are evaluated after 2 years of exposure at CCAFS. Using ASTM D1654 ratings, success is determined similarly to the accelerated corrosion chamber tests. CRS substrates must achieve a rating of greater than or equal to 6 for creep-from-scribe, and greater than or equal to 7 for blisters in field. Al substrates rating greater than or equal to 8 for creep-from-scribe, and greater than or equal to 7 blisters in field. The multimetal specimens rating greater than or equal to 6 for the scribed area, and for area away from the scribe, greater than or equal to 7 combined rating for blisters in field.

Table 18 contains all ratings for CRS taken at 6-month intervals. After 2 years total outdoor exposure only 2 pretreatments—Bonderite 7400 and the NCP nonchromate wash primer—were able to meet the success criteria with both MIL-DTL-53022 and MIL-DTL-53030. The Bonderite 7400 and NCP also rated a 10 for blisters in field, far exceeding the required rating of 7. The consistent performance of the Bonderite 7400 in laboratory tests indicated that it would perform well in outdoor exposure. However, the performance of the NCP product here was unexpected considering its weak performance in earlier accelerated corrosion tests.

Table 18 **ASTM D1654 ratings for outdoor exposure testing on CRS panels**

		Exposure Time (years)							
		Creep from Scribe				Blisters in Field			
Pretreatment		0.5	1	1.5	2	0.5	1	1.5	2
Picklex 20	53022	10	3.4	3	1.8	10	6.8	5.4	4
	53030	7.8	4.6	3.2	3.2	8	8	8	8
SurTec 650	53022	10	3	2.2	1.2	10	10	10	10
	53030	10	6	5	4.6	10	10	10	10
Aero-Green AC-10	53022	9.6	5.2	3.2	2.8	10	9.4	9.4	9
	53030	10	4.8	3.6	3.4	10	9.4	9.4	9.4
PPG 11-TGL-07-Z	53022	10	4	3.7	2.7	10	10	10	10
	53030	10	6.5	5.75	4.5	10	10	10	10
Oxilan 9810/2	53022	10	0.8	0.8	0.5	10	10	10	10
	53030	10	6.6	3	3	10	10	10	10
Bonderite 7400	53022	10	8.6	7.3	7.3	10	10	10	10
	53030	10	8.6	7.0	6.4	10	10	10	10
Ecosil Eco 5-1	53022	10	4.5	3.5	3.3	10	9.6	9.6	9.4
	53030	10	8	7.6	6.8	10	9.6	9.6	9.6
NCP WP N-8237-2.5 A/B	53022	10	7.6	6.8	6.8	10	10	10	10
	53030	10	7	6	6	10	10	10	10
DOD-P-15328 WP	53022	10	4.4	3.4	2.2	10	10	10	10
	53030	10	5.8	3	2.6	10	10	10	10

Also surprising was the inadequate performance of the baseline wash primer on CRS. The baseline wash primer could not meet the success criterion for creep-from-scribe after only 1 year of exposure. This was not in agreement with much of the baseline wash primer's performance on CRS in accelerated corrosion tests. It is interesting that nonchromate pretreatments are being required to meet a standard that the legacy baseline wash primer cannot.

Figures 37–40 are presented as a visual comparison of the Bonderite 7400 versus the baseline wash primer on CRS test panels. These panels do not have an abrasive blasted profile and are a relatively smooth-mill finish. Bonderite 7400 clearly performed better than the baseline with MIL-DTL-53022. It is not as clear on the MIL-DTL-53030 samples, but there is far more undercutting of the coating along the scribe on the baseline wash primer samples.



Fig. 37 **Bonderite 7400 on CRS primed with MIL-DTL-53022 after 2 years**



Fig. 38 Bonderite 7400 on CRS primed with MIL-DTL-53030 after 2 years



Fig. 39 DOD-P-15328 on CRS primed with MIL-DTL-53022 after 2 years



Fig. 40 DOD-P-15328 on CRS primed with MIL-DTL-53030 after 2 years

In general, many of the pretreatments performed adequately throughout the 2 years of outdoor exposure on Al substrates, with Picklex being the exception. The coating system on Picklex samples with MIL-DTL-53022 completely delaminated from the substrate after 1 year (Fig. 41). All of the ratings for Al panels are presented in Table 19. Although all of the pretreatment products did not meet the success criteria of greater than or equal to 8 for scribed area and greater than or

equal to 7 for blisters in field, many did provide at least some benefit. The Bonderite 7400, a consistent performer thus far, barely missed meeting the success criteria with a 7.8 rating for scribed areas with MIL-DTL-53022; but exceeded the requirement with MIL-DTL-53030 and for blisters in field. It is important to note that the Bonderite product rated a 7.8 after 1 year but did not degrade any further through year 2. It can be argued that the Bonderite 7400, with error factored in, could be considered meeting the success criteria in this case.

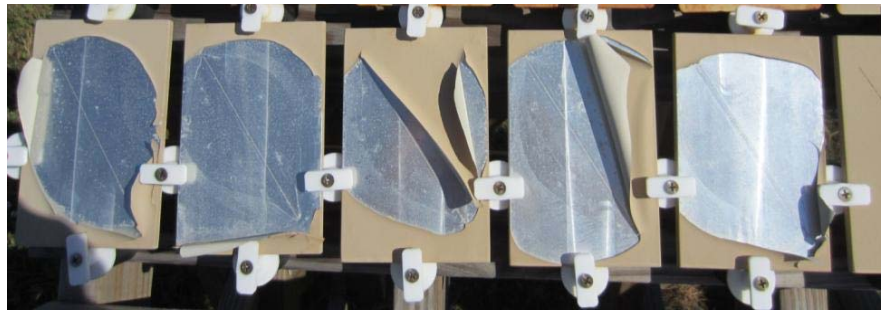


Fig. 41 Picklex on Al primed with MIL-DTL-53022 after 1 year

Table 19 ASTM D1654 ratings for outdoor exposure testing on Al panels

Pretreatment		Exposure Time (hours)							
		Creep from Scribe				Blisters in Field			
		0.5	1	1.5	2	0.5	1	1.5	2
Picklex 20	53022	8	0	0	0	10	0	0	0
	53030	10	7.2	6.6	5.6	10	6.8	6.2	4
SurTec 650	53022	10	9	9	9	10	10	10	10
	53030	10	9	9	9	10	10	10	10
Aero-Green AC-10	53022	10	8.2	7.8	7.2	10	9.4	8.6	7.2
	53030	10	8.3	6.8	5.3	10	9.4	8	5.8
PPG 11-TGL-07-Z	53022	10	9	9	9	10	10	10	10
	53030	10	8.8	8.8	8.8	10	10	10	10
Oxsilan 9810/2	53022	10	9	9	9	10	10	10	10
	53030	10	9	9	9	10	10	10	10
Bonderite 7400	53022	10	7.8	7.8	7.8	10	10	10	10
	53030	10	9	9	9	10	10	10	10
Ecosil Eco 5-1	53022	10	9	8.6	8.4	10	9.6	9.6	8.2
	53030	10	9	9	9	10	9.6	9.4	8.6
NCP WP N-8237-2.5 A/B	53022	10	8.6	8.6	8.4	10	10	10	10
	53030	10	9	9	9	10	10	10	10
DOD-P-15328 WP	53022	10	8.8	8.8	8.8	10	10	10	10
	53030	10	9	9	9	10	10	10	10

Figures 42–45 are presented as a visual comparison of the Bonderite 7400 versus the baseline wash primer on Al test panels. Small areas along the scribe were detected with the retical and measured to arrive at the ratings. However, no significant difference can be discerned visually.



Fig. 42 Bonderite 7400 on Al primed with MIL-DTL-53022 after 2 years

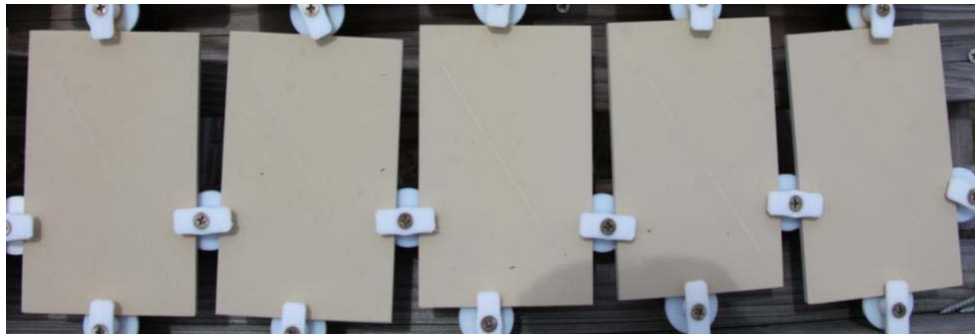


Fig. 43 Bonderite 7400 on Al primed with MIL-DTL-53030 after 2 years



Fig. 44 DOD-P-15328 on Al primed with MIL-DTL-53022 after 2 years

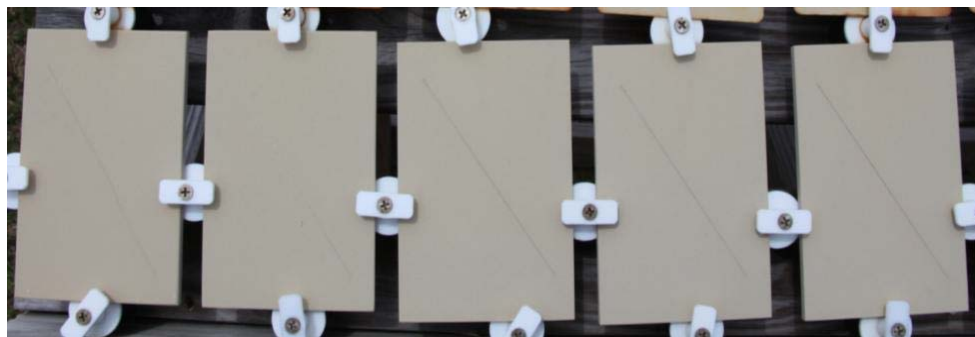


Fig. 45 DOD-P-15328 on Al primed with MIL-DTL-53030 after 2 years

Overall, the multimetal panels fared very well in outdoor exposure, even with the welds and the galvanic couples created with the fasteners and the extruded Al bracket. In general, if coating delamination occurred, it tended to be on the Al “L” brackets. Several of the NCP nonchromate wash primer panels also had delamination along the weld lines. This is reflected in the blisters in field ratings in Table 20. Some blistering was common in and on the U-weld channels, which were meant to collect water. At 2 years, the baseline wash primer was the only pretreatment unable to meet the success criterion for creep from the scribe when primed with MIL-DTL-53030. With MIL-DTL-53022, the baseline wash primer as well as the Oxsilan 9810/2 could not meet the success criterion for the scribed area. However, the baseline was able to meet the criterion for blisters in the field. After examining all results, the Bonderite 7400 with evidential measures has emerged as a viable replacement for chromate wash primer DOD-P-15328. The Bonderite 7400 has shown comparable performance in virtually all testing and has exceeded the performance of the baseline wash primer and met the success criteria in most cases.

Table 20 **ASTM D1654 ratings for outdoor exposure testing on multimetal panels**

		Exposure Time (years)							
		Creep from Scribe				Blisters in Field			
Pretreatment		0.5	1	1.5	2	0.5	1	1.5	2
Picklex 20	53022	8	7.8	7.8	7.4	10	8	7.2	7.2
	53030	9.8	8.8	8	7.3	10	8	6.5	6.5
SurTec 650	53022	10	9	7.8	7	10	8	6	6
	53030	9.8	9	7.2	6.8	10	8	6.2	6.2
Aero-Green AC-10	53022	7	6.8	6.8	6.8	10	8	6.6	6.6
	53030	10	8.6	8	7.2	10	8	7	7
PPG 11-TGL-07-Z	53022	9.6	8.8	7.8	6.6	10	8	7	7
	53030	10	9	7.6	6.4	10	8	7	7
Oxsilan 9810/2	53022	9.2	8.2	6	5.4	10	8	6.4	6.4
	53030	9.4	9	7.6	6.8	10	8	7	7
Bonderite 7400	53022	10	8.8	8.4	7.4	10	8	7.4	7
	53030	10	9	9	7	10	8	7.8	7
Ecosil Eco 5-1	53022	8.8	8.6	8.6	6.4	10	8	7	7
	53030	10	9	8.6	6.6	10	8	7.4	7
NCP WP N-8237-2.5 A/B	53022	8.4	8	7.8	6.6	10	8	7.2	6.2
	53030	8.6	8.4	8.4	7.8	10	8	6.6	6.6
DOD-P-15328 WP	53022	9.4	8.8	6.2	5.8	10	8	7.8	7
	53030	10	8.2	4.8	4.8	10	8	8	7
Direct to Metal	53022	10	9	7.6	6.4	10	8	6.8	6.8
	53030	9.6	8.8	7.8	6.6	10	8	7.8	7

4. Limited Scale Demonstration

Mine-Resistant Ambush Protected (MRAP) Vehicle Doors

Two spare MRAP doors were obtained from the Program Management Office (PMO) and processed at paint facilities at Aberdeen Proving Ground (APG), Maryland. Once processed, the doors were placed in local outdoor exposure

testing at APG. Each door had the window masked off and was abrasive blasted to a 1.5-mil finish before processing. One door was treated with the baseline wash primer DOD-P-15328 and the other was treated with Oxsilan 9810/2. The Oxsilan 9810/2 was selected for demonstration based on early experimental results and the results leveraged from ESTCP project WP 200906.²² A representative from Chemetall, the manufacturer, was present for the Oxsilan pretreatment process and the following procedures were followed:

Chemetall Oxsilan 9810/2:

- 1) Pressure wash all parts to remove dirt and grime.
- 2) Abrasive blast to 1.5 Surface Profile IAW SSPC SP 10.
- 3) Blow-down dust.
- 4) Apply Oxsilan 9810/2 at 70–80 °F.
- 5) Allow 60–90 s contact time.
- 6) Rinse with clean with DeIonized (DI) water and blow dry.
- 7) Apply CARC system after completely dry.

After the pretreatment was fully dry, the doors were primed and painted simultaneously with MIL-DTL-53022 Type II primer and topcoated with MIL-DTL-53039. After curing, the doors were transported to ARL Building 4600 where they were placed on outdoor exposure test racks beginning in August of 2012. This outdoor exposure site lacks an individual weather station; therefore, specific weather data was not collected. However, APG can provide weather data when requested.



Fig. 46 (Left) MRAP door being treated with Oxsilan 9810/2; (Right) Painted doors on outdoor exposure racks at ARL

The Oxsilan 9810/2 performed well in laboratory tests as part of the ESTCP program WP 200906 and was a major reason for considering it as a potential alternative to DOD-P-15328. Although not considered a “full-scale” demonstration, the MRAP doors provide some insight into the processing of larger parts in a production environment. Unlike the wash primer, this product requires a rinsing step following a dwell or contact period to remove the surplus product. In a small-scale repair scenario, this may not be practical. It is more appropriate for larger scale production where a recirculating system can be employed and waste product is captured and reused or removed. Nevertheless, Oxsilan 9810/2 has demonstrated good corrosion resistance and adhesion for abrasive blasted steel substrates. Figures 47 and 48 show the 2 MRAP doors after 2 years outdoor exposure at APG. Understandably, APG is not as aggressive as Cape Canaveral; however, the relative performance of the Oxsilan 9810/2 versus the baseline wash primer can still be assessed. Table 21 lists the ASTM D1654 ratings over the 2 years; however, there is no difference in the corrosion present on the doors or along the scribes as yet. Exposure at APG will likely take longer for significant corrosion to occur.



Fig. 47 MRAP door with Oxsilan 9810/2 after 2 years with close-up of scribed area



Fig. 48 MRAP door with DOD-P-15328 WP after 2 years with close-up of scribed area

Table 21 ASTM D1654 ratings for outdoor exposure testing on MRAP doors

		Exposure Time (years)							
		Creep from Scribe				Blisters in Field			
Pretreatment		0.5	1	1.5	2	0.5	1	1.5	2
Oxsilan 9810/2	53022	10	9	9	9	10	10	10	10
DOD-P-15328 WP	53022	10	9	9	9	10	10	10	10

5. Conclusions

The Bonderite 7400 has been qualified as Type IV pretreatment—class A, B, and C IAW TT-C-490F. If seeking qualification for inclusion on the QPD for TT-C-490F, the current DOD-P-15328 wash primer would not meet the requirements and would not qualify for many of the applications as tested. Additionally, the results presented here show that there are viable alternatives that can provide comparable, and in some cases superior performance to the legacy DOD-P-15328 wash primer. The Bonderite 7400 is one of the products that compares very well with the baseline wash primer and proves to be a viable drop-in replacement. For all of the products tested, a deoxidizing step is advised to improve performance on Al substrates. For applications where deoxidizing of Al substrates is not practical, wash primer, SurTec 650, and the Bonderite 7400 could be used.

The Ecosil 5-1 is not yet commercially available, but the results show promise. The product performed admirably and may be a candidate for the QPD once the formulation is optimized. The NCP nonchromate wash primer provided good corrosion performance in outdoor exposure on both CRS and Al test panels. But

the inconsistent performance in the laboratory tests would preclude the NCP product from being approved for use via TT-C-490F.

The purpose for using the 2 primers in this project was to determine if the pretreatments were compatible or incompatible with specific primer types. The focus was to evaluate the performance of alternative pretreatment for wash primer, but it also became evident that the water reducible primer MIL-DTL-53030 Type IV provided better corrosion protection than the solvent borne MIL-DTL-53022 Type II primer in virtually every scenario.

6. References

1. Department of the Army. Army materiel maintenance- maintenance of supplies and equipment. Washington (DC): Headquarters, Department of the Army; 2013. Army Regulation No.: AR 750-1.
2. MIL-DTL-53072. Chemical agent resistant coating (CARC) system application procedures and quality control inspection. Washington (DC): Department of Defense (US); 2003 Jun.
3. Chemical conversion coatings and pretreatments for metallic substrates (base for organic coatings). Washington (DC): Department of Defense (US); 2013 Jan. TT-C-490.
4. MIL-DTL-53022. Primer, epoxy coating, corrosion inhibiting, lead and chromate free. Washington (DC): Department of Defense (US); 2007 Jan.
5. MIL-DTL-53030. Primer coating, epoxy, water based, lead and chromate free. Washington (DC): Department of Defense (US); 2010 May.
6. MIL-DTL-53039. Coating, aliphatic polyurethane, single component, chemical agent resistant. Washington (DC): Department of Defense (US); 2005 Jun.
7. MIL-DTL-64159. Camouflage coating, water dispersible aliphatic polyurethane, chemical agent resistant. Washington (DC): Department of Defense (US); 2002 Jan.
8. DOD-P-15328. Primer (wash), pretreatment (Formula no. 117 for metals). Washington (DC): Department of Defense (US); 1978 Apr.
9. Chesonis KG, Miller, CE. VOC compliant wash primer, Aberdeen Proving Ground (MD): Army Research Laboratory (US); 1994 Sep. Report No.: ARL-MR-190.
10. Young, JJ. Minimizing the use of hexavalent chromium (Cr6+). Washington (DC): Undersecretary of Defense; 2009.
11. ASTM A1008. Standard specification for steel, sheet, cold-rolled, carbon, structural, high-strength low-alloy, high-strength low-alloy with improved formability, solution hardened, and bake hardenable. West Conshohocken (PA): ASTM International; 2013.

12. ASTM A240/A240M-14. Standard specification for chromium and chromium-nickel stainless steel plate, sheet, and strip for pressure vessels and for general applications. West Conshohocken (PA): ASTM International; 2013.
13. FED-STD-151B. Federal test method standard: “metals; test methods” general services administration. Washington (DC): 20407; (1967 Nov 24). 2014 Oct 17.
14. ASTM D3359. Standard test methods for measuring adhesion by tape test. West Conshohocken (PA): ASTM International; 2009.
15. ASTM D3363. Standard test method for film hardness by pencil test. West Conshohocken (PA): ASTM International; 2005.
16. SAE J400. Test for chip resistance of surface coatings. Reaffirmed: 2012 Oct 23.
17. ASTM B117. Standard practice for operating salt spray (fog) apparatus. West Conshohocken (PA): ASTM International; 2011.
18. GM 9540P. Accelerated corrosion test. Detroit (MI): General Motors Engineering Standards; 1997.
19. ASTM D1654. Standard test method for evaluation of painted or coated specimens subjected to corrosive environments. West Conshohocken (PA): ASTM International; 2008.
20. Chapter 30: Seawater. Corrosion tests and standards: application and interpretation. 2nd ed. West Conshohocken (PA): ASTM International; 2005. 363. Print.
21. Travel Weather Averages (Weatherbase). Weatherbase. N.p., n.d. Web. 2014 Oct 2.
22. Kelley J, Braswell T. ESTCP Project 200906 nonchromate, ZVOC coatings for steel substrates on Army and Navy aircraft and ground vehicles: Technology Area 1: demonstration of steel pretreatments on high hard armor steel. Aberdeen Proving Ground (MD): Army Research Laboratory (US).

Appendix A. Observed Corrosion Rate (mils/year) at CCAFS Since 2011

This appendix appears in its original form, without editorial change.

Coupon	1	2	3	Avg	Date Exposed	Date Removed	Days Exposed	1	2	3	Avg	Δm	Corrosion Rate
660F	29.81	29.81	29.81	29.81	11/9/2010	5/17/2011	189	29.28	29.28	29.28	29.28	0.53	1.675323221
661F	29.89	29.89	29.89	29.89	11/9/2010	5/17/2011	189	29.40	29.40	29.40	29.40	0.49	1.560401741
662F	29.97	29.97	29.97	29.97	11/9/2010	5/17/2011	189	29.42	29.42	29.42	29.42	0.55	1.732256798
663F	29.94	29.94	29.94	29.94	11/9/2010	5/17/2011	189	29.48	29.47	29.48	29.47	0.47	1.48343598
664F	29.99	29.99	29.99	29.99	11/9/2010	12/20/2010	41	29.82	29.82	29.82	29.82	0.17	2.488413904
665F	29.91	29.91	29.91	29.91	11/9/2010	2/16/2011	99	29.59	29.59	29.59	29.59	0.32	1.952419132
666F	30.26	30.26	30.26	30.26	11/9/2010	5/17/2011	189	29.53	29.53	29.54	29.53	0.73	2.293157965
667F	30.07	30.07	30.07	30.07	11/9/2010	11/19/2012	741	27.27	27.27	27.27	27.27	2.81	2.26320573
668F	29.74	29.74	29.74	29.74	11/9/2010	11/19/2012	741	27.27	27.27	27.27	27.27	2.47	1.992675197
669F	29.80	29.80	29.80	29.80	11/9/2010	5/17/2011	189	29.06	29.06	29.06	29.06	0.74	2.343765589
670F	29.99	29.99	29.99	29.99	11/9/2010	2/16/2011	99	29.68	29.68	29.68	29.68	0.31	1.861842987
671F	30.02	30.02	30.02	30.02	11/9/2010	12/20/2010	41	29.85	29.85	29.85	29.85	0.17	2.493274088
296C	29.73	29.73	29.73	29.73	2/15/2011	2/23/2012	373	28.81	28.82	28.82	28.82	0.92	1.46699359
297C	29.75	29.75	29.75	29.75	2/15/2011	2/23/2012	373	28.66	28.66	28.66	28.66	1.10	1.755477399
442F	29.91	29.91	29.91	29.91	5/17/2011	11/19/2012	552	27.95	27.95	27.95	27.95	1.96	2.120827315
443F	29.70	29.70	29.70	29.70	5/17/2011	11/19/2012	552	28.19	28.19	28.19	28.19	1.51	1.639625135
513T	29.90	29.90	29.90	29.90	2/23/2012	2/11/2014	719	28.05	28.05	28.05	28.05	1.84	1.531089586
514T	29.84	29.84	29.84	29.84	2/23/2012	2/11/2014	719	27.77	27.77	27.77	27.77	2.07	1.720795591
100S	29.93	29.93	29.93	29.93	11/19/2012	2/11/2014	449	28.74	28.74	28.74	28.74	1.20	1.59147734
101S	29.76	29.75	29.75	29.75	11/19/2012	2/11/2014	449	28.55	28.55	28.55	28.55	1.20	1.601684807
672F	30.05	30.05	30.05	30.05	11/9/2010	12/20/2010	41	29.91	29.91	29.91	29.91	0.14	2.104459415
673F	30.15	30.15	30.15	30.15	11/9/2010	2/16/2011	99	29.81	29.81	29.81	29.81	0.34	2.053059294
674F	29.97	29.97	29.97	29.97	11/9/2010	5/17/2011	189	29.33	29.33	29.33	29.33	0.64	2.028522264
675F	30.29	30.29	30.30	30.29	11/9/2010	11/19/2012	741	28.56	28.56	28.56	28.56	1.73	1.399444227
676F	30.03	30.03	30.03	30.03	11/9/2010	11/19/2012	741	28.23	28.23	28.23	28.23	1.80	1.450538463
677F	30.08	30.08	30.08	30.08	11/9/2010	5/17/2011	189	29.49	29.49	29.49	29.49	0.59	1.86299316
678F	30.01	30.01	30.01	30.01	11/9/2010	2/16/2011	99	29.66	29.65	29.65	29.65	0.35	2.143635439
679F	30.09	30.09	30.09	30.09	11/9/2010	12/20/2010	41	29.92	29.92	29.92	29.92	0.17	2.537015738
298C	29.61	29.61	29.61	29.61	2/15/2011	2/23/2012	373	28.75	28.75	28.75	28.75	0.86	1.373503467
299C	29.75	29.76	29.76	29.76	2/15/2011	2/23/2012	373	28.89	28.89	28.89	28.89	0.86	1.380982677
440F	29.88	29.89	29.89	29.89	5/17/2011	11/19/2012	552	28.50	28.50	28.50	28.50	1.38	1.498116316
441F	29.91	29.92	29.92	29.92	5/17/2011	11/19/2012	552	28.32	28.32	28.32	28.32	1.60	1.732400049
515T	29.89	29.89	29.89	29.89	2/23/2012	2/11/2014	719	27.72	27.72	27.72	27.72	2.17	1.8069878
516T	29.73	29.72	29.72	29.72	2/23/2012	2/11/2014	719	27.82	27.82	27.82	27.82	1.91	1.584717215
512T	29.92	29.92	29.92	29.92	2/23/2012	2/11/2014	719	27.74	27.74	27.74	27.74	2.17	1.806433509
519T	29.89	29.89	29.89	29.89	2/23/2012	2/11/2014	719	28.02	28.02	28.02	28.02	1.87	1.553676934
104S	29.82	29.82	29.82	29.82	11/19/2012	2/11/2014	449	28.47	28.47	28.47	28.47	1.36	1.804502751
105S	29.94	29.94	29.94	29.94	11/19/2012	2/11/2014	449	28.71	28.71	28.71	28.71	1.23	1.63674524
												Avg rate	1.824365186

Appendix B. Weather Data for 2012–13 at CCAFS Site

This appendix appears in its original form, without editorial change.

Values							
Row Labels	Sum of Solar		Average of Temp, °F	Average of RH, %	Average of DewPt, °F	Average of Wetness, Sum of Time of	
	Sum of Rain, in	Radiation, W/m²				%	Wetness, hr
Jan	0.00	375,267.70	62.6	76.4	54.2	56.1	503.3
Feb	0.92	435,153.20	68.6	79.9	61.6	58.0	479.5
Mar	0.83	742,881.40	73.5	74.2	64.1	60.6	509.8
Apr	0.74	801,708.10	75.6	69.5	63.9	51.6	446.8
May	2.96	819,811.80	79.7	77.4	71.4	58.5	547.0
Jun	6.80	692,588.30	80.9	79.7	73.5	57.0	556.8
Jul	3.58	878,208.60	83.5	82.6	77.3	65.8	654.3
Aug	6.67	675,046.90	81.9	86.8	74.9	74.8	734.5
Sep		634,962.40	81.1	81.9	70.6	74.7	720.0
Oct		508,110.60	76.7	77.5	69.6	68.6	744.0
Nov		353,967.40	65.9	80.7	67.1	69.3	715.8
Dec		298,045.20	66.5	83.0	60.6	80.5	706.8
Grand Total	22.50	7,215,751.60	74.7	79.1	67.4	64.7	7318.3

Appendix C. Weather Data for 2013–14 at CCAFS Site

This appendix appears in its original form, without editorial change.

Values							
Row Labels	Sum of Solar		Average of Temp, °F	Average of RH, %	Average of DewPt, °F	Average of Wetness, Sum of Time of	
	Sum of Rain, in	Radiation, W/m²				%	Wetness, hr
Jan	0.00	308,732.90	67.6	85.5	62.7	84.9	738.3
Feb	1.06	464,236.70	66.2	77.6	58.1	69.0	609.8
Mar	0.84	717,370.30	62.5	69.4	50.9	58.6	647.3
Apr	4.19	706,461.70	75.1	81.1	68.4	78.9	720.0
May	6.28	781,948.20	77.4	76.1	68.6	62.3	642.0
Jun	4.28	733,323.10	82.6	83.1	76.6	54.1	444.0
Jul	3.92	724,836.80	80.8	87.7	76.5	56.4	454.8
Aug	2.06	767,085.70	84.2	82.0	77.8	44.1	394.5
Sep	1.58	625,460.50	82.0	79.9	74.8	42.1	396.0
Oct	2.88	546,972.00	77.3	79.4	69.9	39.3	363.0
Nov	0.88	295,750.70	72.2	81.7	65.9	64.8	581.8
Dec	0.22	278,694.70	69.3	85.9	64.5	71.0	628.3
Grand Total	28.19	6,950,873.30	74.8	80.8	67.9	60.4	6619.5

List of Symbols, Abbreviations, and Acronyms

Al	Aluminum
APG	Aberdeen Proving Ground, Maryland
AR	Army Regulation
ARL	US Army Research Laboratory
ASTM	American Society for Testing and Materials
ATC	Aberdeen Test Center
CARC	Chemical Agent Resistant Coating
CCAFS	Cape Canaveral Air Force Station
CCPE	Corrosion Control and Prevention Executive
COTS	commercial-off-the-shelf
CRS	cold rolled steel
DI	DeIonized
DOD	Department of Defense
DTM	direct-to-metal
ESTCP	Environmental Security Technology Certification Program
GM	General Motors
h	hour(s)
IAW	in accordance with
min	minute(s)
MRAP	Mine-Resistant Ambush Protected
OSD	Office of the Secretary of Defense
PEO	Program Executive Office
PMO	Program Management Office
QPD	qualified product database
s	second(s)

TACOM	Tank Automotive and Armaments Command
VOC	Volatile Organic Compound
WP	Weapons Platforms

1 DEFENSE TECHNICAL
(PDF) INFORMATION CTR
DTIC OCA

2 DIRECTOR
(PDF) US ARMY RESEARCH LAB
RDRL CIO LL
IMAL HRA MAIL & RECORDS
MGMT

1 GOVT PRINTG OFC
(PDF) A MALHOTRA

1 DIR USARL
(PDF) RDRL WMM C
J KELLEY

INTENTIONALLY LEFT BLANK.